Scottish Universities Environmental Research Centre

DEVELOPMENT OF PROFICIENCY TESTING FOR DETECTION OF IRRADIATED FOOD PROJECT E01068

FINAL REPORT NOVEMBER 2007

VOLUME III: RESULTS OF THIRD ROUND PSL/ TL TRIALS JUNE 2007

D.C.W. Sanderson, L.A. Carmichael, S. Fisk, P. Key, E.M. Scott

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D.C.W. Sanderson, L.A. Carmichael, S. Fisk, P. Key, E.M. Scott

Participants :

Angelo Alberti, Stephan Barth, Rainer Brockmann, Peter Brown, Joanne Chan Sheot Harn, Rupa Das, Frank Dittmar, Esko Niemi, Airi Paajanen, Sandro Onori, Emanuela Bortolin, Juergen Pfordt, Nicola Sardone, Irene Straub, Heidi Flaschka, Setsuko Todoriki, Claus Wiezorek, Meike Bergmann, Magda Nuchter-Frangos, Andy Ward, Irene Poulima, Wolfgang Kruspe, Christine Scleich, Jurgen Brunner, Beate Muller, Brigitte Butz, Deirdre Murphy, Gary Ridgewell, Michael Sunderland, Helen Davies, Brenda Lennon, David Johnson, Chiaravalle Antonio Eugenio, Bert Popping, Angela Li, Nicolas Feuillere, Christopher Young, Benoit Le Gall, Julien Senez, Reena Kiriyanthan, Alan Bruce, Gary Walker, Ryang Jun Hwan, Kim Byeong Keun, Dongmi Choi, John Hill, Jack Garfoot, Robbie Beattie, Grzegorz Guzik, K.Malec-Czechowska, G.Liśkiewicz, M.Laubsztejn, Darren Clark.

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SUMMARY

This report summarises the results from Round 3 in which participants used the BS EN 13751:2002 photostimulated luminescence (PSL) method and the BS EN1788:2001 thermoluminescence (TL) method. Recognising the fundamentally qualitative nature of irradiation testing methods and the need to examine the relationship between underlying quantitative data and their associated qualitative outcomes led to a trial which utilised large numbers of test materials in Rounds 1 and 2, and a much-reduced set in Round 3. Participant numbers have increased over the project, with 32 data sets returned in round 1, increasing to 45 in round 2 and to 51 data sets in this round, comprising 35 PSL returns and 16 TL laboratories. For TL, sample numbers were the same in the 2 rounds using this technique.

In this third round, 18 newly procured test materials of herbs, spices, seasonings and dietary supplement ingredients were prepared in irradiated and unirradiated forms and as blends. The selection aimed to include a range of sensitivities (from low to high), which together with 3 different blend concentrations would again test proficiency at the limits of the method. The samples selected for the study comprised three herbs and spices and three dietary supplement ingredients, presented untreated, irradiated and as blended mixtures. One of these samples, a ginseng root product, was subsequently shown to be carrying high PSL signals, and associated TL signals in its untreated form. It has been included in the study, but cannot be considered to be unirradiated material on the basis of the data gathered during reference analyses and by participants. The supplier and the Food Standards Agency were notified of this finding.

35 participating laboratories conducted PSL determinations and measurements from standard materials and returned results to the SUERC; 10 also carried out calibrated PSL determinations. 16 laboratories participated in TL separations and measurements from the same 18 samples. Additional material for each sample was provided to those laboratories which conducted both methods.

Reference data for the PSL screening part of the study comprised values obtained from the 18 materials, from 10 pots of each sample, analysed in duplicate. Screening measurements were performed in parallel on two machines, one the system used for Rounds 1 and 2, the other utilising a recently developed Windows-XP-based PSL system which is completing trials. Calibrated PSL was performed on the samples screened on the DOS system. Thus an additional 1080 PSL reference analyses were conducted in support of round 3. For the 18 TL samples 20 separations per sample were performed, followed by TL analysis, comprising 360 single aliquot TL determinations, equivalent to 180 EN1788 duplicated analyses. The reference analyses are documented in this report and used, where appropriate, to define assigned values for *z*- score determination.

Participants returned results for PSL screening, calibrated PSL and TL analyses in a timely manner. The PSL screening results once again produced high quality results. There are no longer any laboratories with conspicuous sample handling difficulties. Quantitative analyses based on *z*-scores were again able to reveal both sample and laboratory specific differences in behaviour but these are less marked than in earlier rounds. The identification and remediation of such problems continues to be a significant achievement for PT work of this sort, and underlines its importance.

Calibrated PSL data were returned by 10 laboratories, each of whom succeeded in producing data sets that conformed to EN13751, despite having diverse access to irradiation facilities. Based on comments returned by the participants, however, most of them did not utilise the additional information derived from calibration to modify their evaluations. New approaches to analysis of

calibrated PSL data sets, based on regression analysis have been developed and will be used to evaluate these interlaboratory data sets.

For the TL study 16 participants returned data. Participants were offered a choice between analysing all 18 samples and using a reduced of 9 (specified), following the questionnaire distributed after Round 2. TL data are again presented here both using z score analysis of the glow ratios, and a quatitative analysis of the classification outcomes. As before the outcomes are broadly successful, although on this occasion there is some evidence of interlaboratory performance differences, including a minority of laboratories where z scores from unirradiated mqaterials suggest the possibility of cross contamination. Since classification of TL data in based on a combination of glow ratio and presence of low temperature signal components, qualitative approaches to performance evaluation seem potentially preferable. However the cost and timescales for TL analysis necessarily preclude the use of the large sample numbers available from PSL data sets in earlier rounds. In this round qualitative assessment based on participants' comments showed that all the irradiated materials were correctly identified as such. More than 80% of unirradiated materials were correctly identified but for the blends fewer than 40% were classified as mixtures. These samples clearly presented more difficulty than the "pure" products, which is not unexpected, particularly since the concentration-sensitivity relationship of the blends made some of them quite challenging A new approach to assessing interlaboratory performance has also been explored briefly in this round, based on evaluation of the ratio of first glow signal intensities with the laboratory minimum detectable level. This has the advantages of being independent of the units used to record TL data, but responds to a combination of laboratory blank, efficiency of mineral recovery during sample preparation and intrumental sensitivity. Based on initial examination in this study, this appears to be a sensitive indicator of interlaboratory differences and therefore may be useful in future proficiency testing analyses of TL results.

Overall the third round of the FSA proficiency testing project has again produced a wealth of useful results. After three rounds of PSL screening trials it seems that participants' performance has steadily improved, particularly in respect of sample handling and avoidance of cross-contamination. There are sensitivity differences between individual laboratories, borne out by response to the paprika standards and by pooled *z* -scores for irradiated samples. The correlations observed between these indicators and qualitative classification outcomes suggest that pooled *z* -scores would be an effective way of scoring PSL proficiency. On this basis it would be possible to implement a routine scheme based on relatively modest numbers of samples. Calibrated PSL has been applied now for the second time, and a new approach to comparison of results, based on regression analysis has been proposed. It appears that this may also be appropriate to classification of laboratory performance, although further work would be needed to assess whether sparse data sets such as the 18 sample design used here, are sufficient to define robust regression parameters for routine PT scheme use. For TL this round and the preceding round have generally shown that participating laboratories are able to generate broadly comparable outputs. Detailed examination of *z*-scores from glow ratios of unirradiated samples, and of the signal to background ratios associated with first glow peak intensities, reveals performance differences which may merit further attention in future rounds. The qualitative outcomes are encouraging, but are limited at this stage by the relatively small numbers of samples and laboratories. It is to be hoped that this work will continue, since it is generating valuable data that are making a useful contribution to enhancing overall performance in laboratories applying EN13751 and EN1788 analysis, and to assuring users of these data that good analytical performance can be achieved across a community of participating laboratories.

ACKNOWLEDGMENTS

Support for this work by the Food Standard Agency, through project E01068, is gratefully acknowledged. Materials were purchased from Schwarz McCormicks UK and Cambridge Commodities. Irradiation of test materials took place at Isotron plc and of homogeneity testing samples at the Beatson Centre for Oncology. We are also grateful to the following participants for the work and time they have contributed to the third round of this study:

Angelo Alberti, Stephan Barth, Rainer Brockmann, Peter Brown, Joanne Chan Sheot Harn, Rupa Das, Frank Dittmar, Esko Niemi, Airi Paajanen, Sandro Onori, Emanuela Bortolin, Juergen Pfordt, Nicola Sardone, Irene Straub, Heidi Flaschka, Setsuko Todoriki, Claus Wiezorek, Meike Bergmann, Magda Nuchter-Frangos, Andy Ward, Irene Poulima, Wolfgang Kruspe, Christine Scleich, Jurgen Brunner, Beate Muller, Brigitte Butz, Deirdre Murphy, Gary Ridgewell, Michael Sunderland, Helen Davies, Brenda Lennon, David Johnson, Chiaravalle Antonio Eugenio, Bert Popping, Angela Li, Nicolas Feuillere, Christopher Young, Benoit Le Gall, Julien Senez, Reena Kiriyanthan, Alan Bruce, Gary Walker, Ryang Jun Hwan, Kim Byeong Keun, Dongmi Choi, John Hill, Jack Garfoot, Robbie Beattie, Grzegorz Guzik, K.Malec-Czechowska, G.Liśkiewicz, M.Laubsztejn, Darren Clark.

1. INTRODUCTION

Irradiation is used in many countries for the purposes of shelf life extension, reduction of spoilage and pathogen content, and retardation of ripening and sprouting processes in many different foods. UK[1,2,3,4] and European Regulations[5,6] require both licensing of plant and process and explicit product labelling at all stages of market presentation. However, in the absence of widespread consumer acceptance, there is little evidence of properly labelled products in the UK or in Europe. Several analytical methods for detection of food irradiation have been developed, of which the CEN international standards [7,8,9,10,11,12,13,14,15,16] (i.e. within the UK the BS EN series) based on luminescence are in quite widespread use. Both photostimulated luminescence (PSL) screening and thermoluminescence (TL) analysis were used successfully in the 1996 MAFF survey of undeclared foods[17] and also in the 2001 survey conducted by the Food Standards Agency[18]. These surveys have been successful in identifying undeclared irradiated spices and shellfish and particularly in drawing attention to the significant problems associated with dietary supplements[19], which are the subject of current enforcement actions in the UK, and elsewhere in Europe. With this in mind the Food Standards Agency commissioned this project to assess the feasibility of developing a proficiency scheme appropriate for the detection of irradiated foods.

International Harmonised Protocols for conducting proficiency testing of analytical methods and laboratories are available[20,21], and form the basis for schemes such as those operated by

[1] Food (Control of Irradiation) Regulations, 1990, SI 2490

[2] The Food Irradiation Provisions (England) Regulations 2000, 2000,SI 2254

[3] The Food Irradiation Provisions (Wales) Regulations 2001, 2001, WSI 1232 (W.66)

[4] The Food Irradiation Provisions (Scotland) Regulations 2000, 2000, SSI 309

[5] European Directive 1999/2/EC, On approximation of the laws of the Member States concerning foods and food ingredients treated with Ionising Radiation, OJEC, February 1999

[6] European Directive 1999/3/EC, On the establishment of a community list of foods and food ingredients treated with ionising radiation, OJEC, February 1999

[7] BS EN 1784:1996 Foodstuffs - Detection of Irradiated Food Containing fat – Gas Chromatographic Analysis of Hydrocarbons

[8] BS EN 1785:1996 Foodstuffs - Detection of Irradiated Food Containing fat – Gas Chromatographic/Mass Spectrometric Analysis of Alkylcycclobutanones

[9] BS EN 1786:1996 Foodstuffs - Detection of Irradiated Food Containing Bone – Method by ESR Spectroscopy

[10] BS EN 1787:2000 Foodstuffs - Detection of Irradiated Food Containing Cellulose by ESR Spectroscopy

[11] BS EN 13708:2001 Foodstuffs - Detection of Irradiated Food Containing Crystalline Sugar by ESR Spectroscopy

[12] BS EN 13783:2001 Foodstuffs - Detection of Irradiated Using Direct Epifluorescent Filter technique/Aerobic Plate count (DEFT/APC) – Screening Method

[13] BS EN 13784:2001 Foodstuffs - DNA Comet Assay for the Detection of Irradiated Foodstuffs – Screening method

[14] BS EN 1788:1997 Foodstuffs - Detection of Irradiated Food From Which Silicate Minerals can be Isolated: Method by Thermoluminescence

[15] BS EN 13751:2000 Foodstuffs - Detection of Irradiated Food Using Photostimulated Luminescence

[16] BS EN 14569:2004 Foodstuffs – Microbiological Screening for Irradiated Food Using LAL/GNB Procedures.

[17] MAFF, 1997, Undeclared Irradiation of Foodstuffs Surveillance Exercise, Food Surveillance Information Sheet, 102

[18] Food Standards Agency 2002, Survey for Irradiated Foods – Herbs and Spices, Dietary Supplements and Prawns and Shrimps, Food Survey Information Sheet 25/02

[19] European Commission, 2002, Report from the Commission on Food Irradiation for the period September 2000 to December 2001, OJEC, 255,2-12

[20] Thompson M., and Wood R, 1993,The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories, IUPAC/ISO/AOAC Protocol for Proficiency Testing, IUPAC, Geneva

FAPAS® and others for quantitative analysis of foods. However as noted in the earlier report on the first PT trial round [22] there are methodological questions concerning the adaptation of quantitative PT approaches to the qualitative criteria of irradiation tests.

In the first round[22], 32 PSL screening laboratories examined 49 samples presented as irradiated and unirradiated pairs. Reference analyses were conducted at SUERC and used to define standard values for evaluation of z scores from participants' data. These were compared with qualitative classifications and it was shown that PSL screening outcomes were closely related to z score performance. Some performance differences between laboratories were noted, and suggestions made for enhancing sample handling integrity in the second round. In discussion with participants it was also agreed to use replicate samples in future rounds to bring the PT protocol into line with routine practice in the laboratories.

In the second round[23], in addition to continuing to study PSL screening, Calibrated PSL measurements and TL analysis were included. PSL screening was conducted by 29 laboratories, measuring 72 samples comprising 33 irradiated and unirradiated pairs, plus 6 blended mixtures of irradiated and unirradiated products. Calibrated PSL results were returned from the same sample sets by 11 participants. The TL analysis tasks involved measurement of 18 samples comprising irradiated, unirradiated and blended mixtures prepared from 6 products. TL analysis was undertaken by 16 laboratories.

In this report the third round PT trial is discussed. The third round followed the design of round 2 but with a reduced suite of samples, all from newly procured materials. 18 samples were presented for both PSL and TL analyses, with the option offered to participants of further reducing the TL analyses to 9 samples. PSL screening was conducted by 35 laboratories, 10 of which also performed Calibrated PSL. 16 laboratories undertook TL, with 7 opting to analyse all 18 samples.

This report outlines the design and preparation of round 3 together with details of the new reference analyses undertaken, and an analysis of participants' results.

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[21] Thompson,M., et al, 2006, The International Harmonized Protocol For The Proficiency Testing Of Analytical Chemistry Laboratories, IUPAC TechniCal Report, in press

[22] D.C.W. Sanderson, L.A. Carmichael, S. Fisk, P. Key, E.M. Scott And M. Thompson, 2005, Development of Proficiency Testing For Detection Of Irradiated Food. Project E01068: Results Of First Round PSL Trials, SUERC, East Kilbride

[23] D.C.W. Sanderson, L.A. Carmichael, S. Fisk, P. Key, E.M. Scott And M. Thompson, 2006, Development of Proficiency Testing For Detection Of Irradiated Food. Project E01068: Results Of Second Round PSL Trials September 2006, SUERC, East Kilbride

2. IMPLEMENTATION OF THIRD ROUND (PSL & TL)

In this section the work undertaken in preparation for round 3 is described. Retained materials from Round 1 were insufficient, therefore further products were procured and further reference analyses undertaken (screening and Calibrated PSL and TL).

2.1 Outline of round 3

Round 3 aimed to build on rounds 1and 2 by repeating all the PSL screening, Calibrated PSL and TL measurements as performed in round 2, but on a reduced suite of samples. Six products were chosen and presented in each of three categories; unirradiated, irradiated and blended.

2.2 Participants

Most of the participants from rounds 1 and 2 indicated that they were willing to proceed to round 3. Additional participants for PSL screening and TL joined the study. The full list of participants is given in Table 2.1 below. New laboratory numbers were allocated for this round, which for those who had participated in earlier rounds are in most cases different. Where the three rounds are being compared, the laboratory numbers are those from Round 3.

Table 2.1. List of Round 3 Participants and their institutions

Name	Address
Angelo Alberti	Consiglio Nazionale delle Ricerche Instituto per la Sintesi Organica ela Fotoreattivita (ISOF-CNR), Bologna, Italy
Stephan Barth	Federal Research Centre of Nutrition & Food (BfEL), Karlsruhe, Germany
Rainer Brockmann	Chemisches und Veterinaruntersuchungsamt Ostwestfalen-Lippe CVUA OWL, Bielefield, Germany
Peter Brown	Lincolne Sutton & Wood Ltd, UK
Joanne Chan Sheot Harn	Centre for Analytical Science, H S A, Singapore
Rupa Das	BI Nutraceuticals, USA
Frank Dittmar	Landesbetrieb Hessisches Landeslabor, Kassel, Germany
Ryang Jun Hwan, Kim Byeong Keun	Nong Shim Co.Ltd, Korea
Brenda Lennon	Public Analyst Laboratory (Galway), Eire
Gary Ridgewell	East Anglian Food Ingredients Ltd, UK
Gary Walker, Alan Bruce	Glasgow Scientific Services, UK
Esko Niemi, Airi Paajanen	Finnish Customs Laboratory, Finland
Sandro Onori, Emanuela Bortolin	Instituto Superiore di Sanita, Rome, Italy
Juergen Pfordt	LAVES - Lebensmittelinstitut Oldenburg
John Hill, Jack Garfoot	British Pepper and Spice Co, Ltd, UK
Nicola Sardone	Indena SpA, Settala, Italy
Irene Straub	Chemisches- und Veterinaruntersuchungsamt, Karlsruhe, Germany
Setsuko Todoriki	National Food Research Institute, Japan
Meike Bergmann, Magda Nuchter-Frangos	Berliner Betrieb fűr Zentrale Gesundheitliche Aufgaben, Institute fűr Lebensmittel, Arzneimittel un Tierseuchen, Berlin, Germany
Andy Ward	Unilever UK Foods, Purfleet, Essex
Deirdre Murphy	Public Analyst Laboratory (Cork), Eire
Irene Poulima	Food Division Laboratory, General Chemical State Laboratory of Greece
Bert Popping	Eurofins Scientific Group, Hamburg, Germany
Wolfgang Kruspe	Thuringer Landesamt fur Lebensmittelsicherheit und Verbraucherschutz (TLLV)
Christine Schleich	Landersuntersuchungsamt, Institut fur Lebensmittelchemie und Arzneimittlprufung Mainz
Jurgen Brunner	LUA Sachsen
Beate Muller	Landeslabor Brandenburg, Standort Frankfurt (Oder) Fachgebiet L4, Labor Bestrahite Lebensmittel
Brigitte Butz	Bayerisches Landesamt fur Gesundheit und Lebensmittelsicherheit
David Johnson	Sensient-Tech, California, USA
Michael Sunderland, Helen Davies	Thompson & Capper Ltd, Cheshire, UK
Chiaravalle Antonio Eugenio	Dipartimento di Chimica, Italy
Nicolas Feuillere, Christopher Young, Benoit Le Gall, Julien Senez	Naturex, France, California and New Jersey USA
Reena Kiriyanthan	NBTY Inc, USA
Robbie Beattie	Edinburgh Scientific Services, UK
Dongmi Choi	Korea Food & Drug Administration(KFDA), Korea
Grzegorz Guzik	Laboratory for Detection of Irradiated Food, Institute of Nuclear Chemistry and Technology, Poland
Claus Wiezorek	Chemisches Landes- und Staatliches Veterinäruntersuchungsamt Münster, Germany
Darren Clark	Cardinal Health 414 Ltd, Swindon, UK

2.3 Test Material Preparation and Handling

2.3.1 PSL Materials

The sample allocation comprised 18 samples from 6 products (which were also used in the TL study). As in round 2, duplicated screening measurements from the sample set were specified in the protocol.
Table 2.2 lists the products used in round 3. Green Tea was substituted for ginseng in the blends after it was discovered that the ginseng spike did not have sufficient sensitivity.

Table 2.2. Round 3 Test Materials List

Test Material No.	Product Description	SUERC Ref No	Status
1	Ground Cinnamon	SP10895	U, B, I
2	Ground Cumin	SP10897	U, B, I
3	Thyme	SP10902	U, B, I
4	Siberian Ginseng	SP10950	U, I
5	Alfalfa	SP10951	U, B, I
6	Green Tea	SP10952	B
7	Guarana	SP10954	U, B, I

The blending process is described in Appendix E.
For each of the new materials 250 pots were purchased. For the dietary supplement ingredients bulk material was also obtained for the spike and matrix of the blends. The pots were numbered from 1 to 250 and then re-arranged in a randomly generated array. Each of the 18 samples was given a random sample number (1to 18) the same numbers were used for the TL samples.

Sample	Description	Status	Number	Sample no.
SP10951	Alfalfa	U	1	10
SP10951	Alfalfa	I	2	11
SP10951	Alfalfa	B	3	4
SP10950	Ginseng	U	4	17
SP10950	Ginseng	I	5	1
SP10952	Green tea	B	6	9
SP10954	Guarana	U	7	14
SP10954	Guarana	I	8	15
SP10954	Guarana	B	9	12
SP10902	Thyme	U	10	16
SP10902	Thyme	I	11	2
SP10902	Thyme	B	12	18
SP10897	Cumin	U	13	3
SP10897	Cumin	I	14	8
SP10897	Cumin	B	15	6
SP10895	Cinnamon	U	16	13
SP10895	Cinnamon	I	17	5
SP10895	Cinnamon	B	18	7

Table 2.3. Round 3 Sample Allocation

2.3.2 TL Materials

All 18 samples were offered to the TL laboratories, but since some had already indicated in the Round 2 questionnaire that they preferred 9 samples, the option was given to return results for a specified subset.

2.3.3 Blending

As in round 2, concentrations of 0.1, 1 and 10% were prepared with one dietary supplement, one herb or spice at each concentration.

Alfalfa and thyme were prepared at 0.1%, green tea and cumin at 1% and guarana and cinnamon at 10%.

2.3.3.1 Mixing process

After the experience of round 2 where elaborate mixing did not result in greater homogeneity, the blends for round 3 were mixed for a shorter period. The paprika standard supplied to each laboratory was not homogenised.

Sample packs were distributed on the 5th April 2007. The protocol which participants were asked to follow is presented in Appendix A.

3. PSL SCREENING

3.1 PSL Homogeneity testing (HT)

In Round 2, it was possible to utilise the homogeneity testing data from Round 1, with the exception of the blends which were only introduced in Round 2. For Round 3, however, a complete new set of materials had had to be purchased, necessitating repeat homogeneity testing for both PSL and TL. In addition, SUERC is currently assessing the performance of a modification to the PSL system to enable it to be controlled by Windows-based software. Therefore two full sets of screening data were obtained (10 pots per sample, analysis performed in duplicate). Histograms are presented in Figure 3.1 for both sets of measurements which show that the two systems are equivalent. One participant also used the Windows system; this laboratory's data have been compared with the DOS-based set of reference analyses.

It is noticeable from both histograms that there are 3 columns representing unirradiated material which are in the region otherwise occupied by irradiated material. During homogeneity testing it was observed that the "unirradiated" ginseng displayed an elevated signal. At that point it was too late to remove it from the study, but wherever this elevated signal has significance for interpretation of the data, this is highlighted in the text.

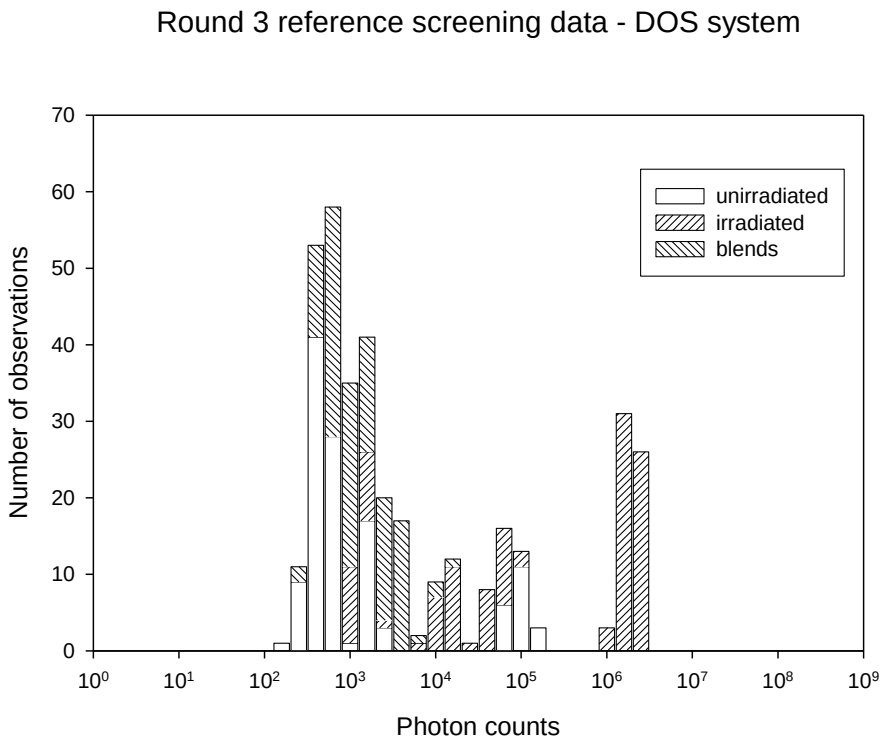
Table 3.1 presents the results of 10 aliquots of paprika standard measured with the 2 systems. Two PSL instruments were used for the comparison, with different sensitivities; test data from the instruments when new indicate that the relative sensitivity derived from the 10 paprika measurements conducted as part of the HT are consistent with the inter-instrument difference. It does not therefore appear that the different software affects the data.

The petri-dishes from the DOS system HT were then irradiated to approximately 1kGy with the ^{60}Co γ-source at the Beatson Centre for Oncology in Glasgow. Reference data for Calibrated PSL were obtained from these aliquots and discussed in section 4 below.

Results for the round 3 measurements of the paprika standard are tabulated below.

Paprika Test Material	DOS System Terminal Counts	Windows System Terminal Counts
1	81547	152471
2	103800	146843
3	89014	154538
4	114256	161996
5	164244	170460
6	99408	166635
7	90670	162849
8	108007	180445
9	149905	147509
10	114980	129871
Mean	111583.10	157361.70
SD	26522.34	14288.05
CV(%)	23.77%	9.08%

Table 3.1. Results of 10 replicate measurements from the paprika reference material, measured by SUERC.



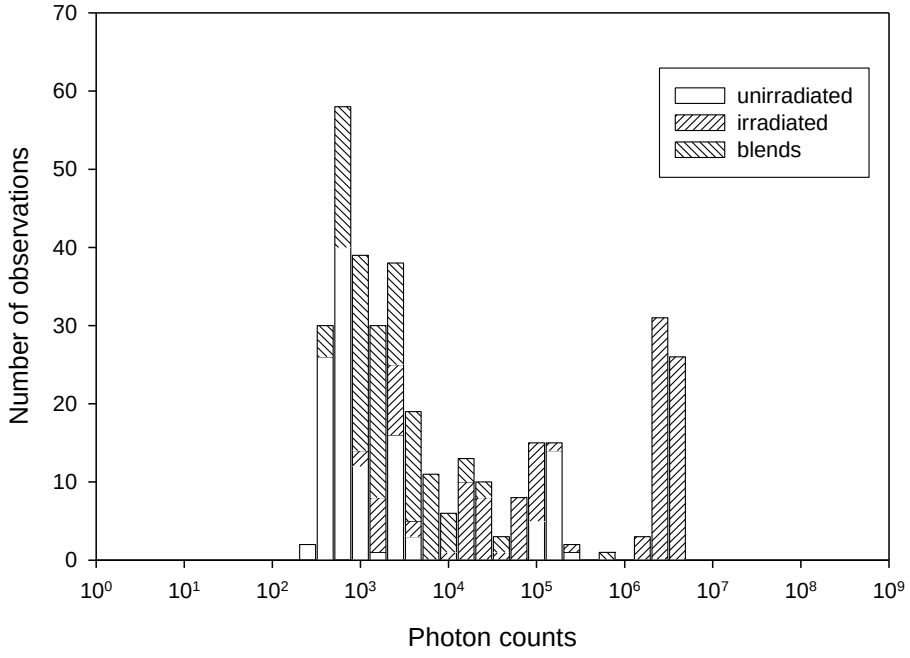


Figure 3.1 Histograms of round 3 reference data for DOS and Windows systems

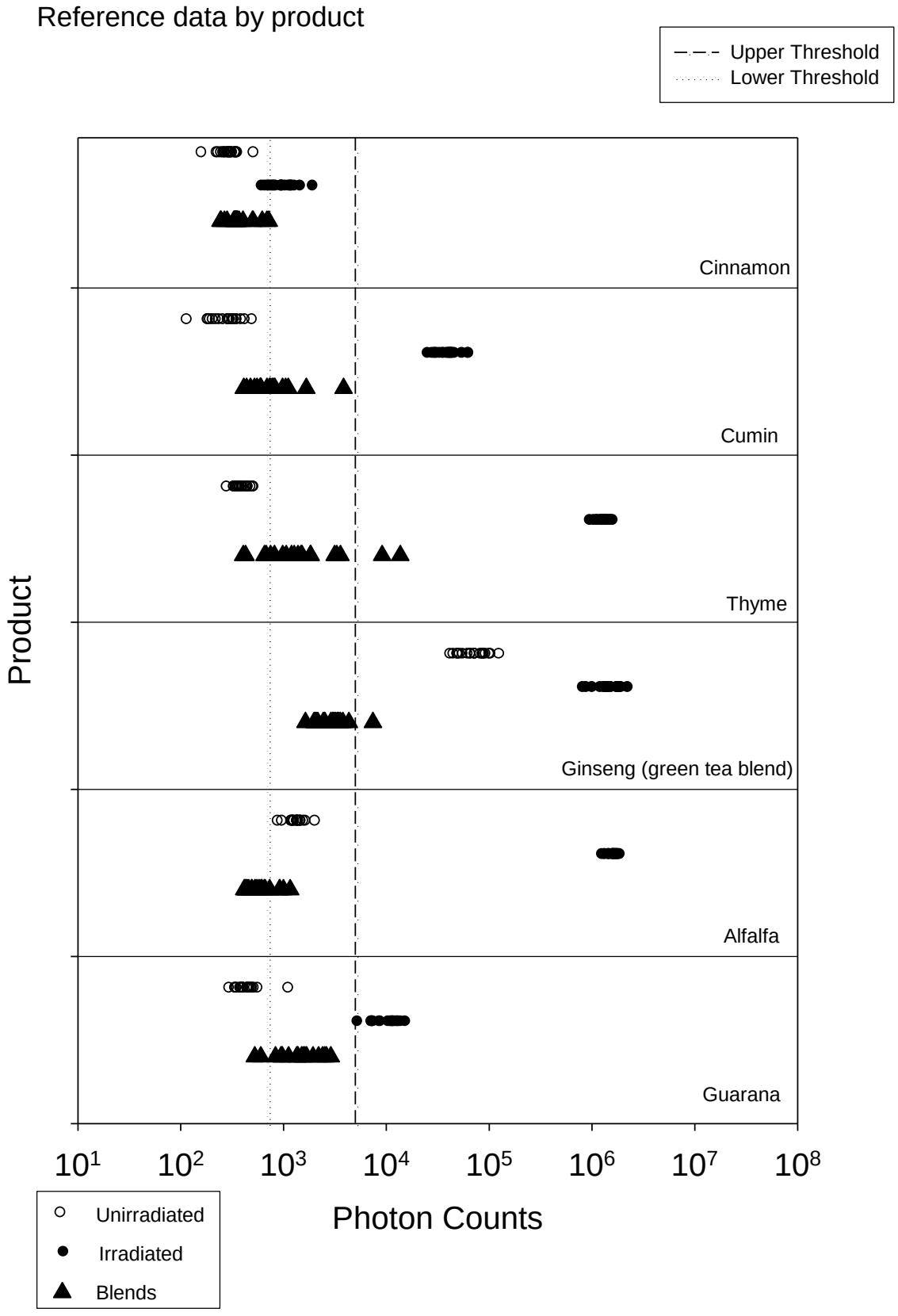


Figure 3.2 Reference data by product

Figure 3.2 presents the new reference data as a log scatter plot for each material in its three categories. Unirradiated samples, with the exception of the ginseng mentioned above, which is entirely above the upper threshold, fall mostly in the negative and intermediate bands. Irradiated samples are with one exception positive, and exhibit marked sample to sample sensitivity variations; the cinnamon is of sufficiently low sensitivity for one observation to lie below the lower threshold. The blended samples are generally located between unirradiated and irradiated results, again as expected. The cinnamon, despite being blended at 10%, the highest concentration, still yields mostly negative results for the blend, reflecting its low sensitivity.

Table 3.2 Summary statistics for reference analysis, DOS and Windows systems with relative sensitivities for each material

		DOS					
		Linear			Log		
		Mean	SD	CV	Mean	SD	CV
Unirradiated							
SP10895	Cinnamon	309.85	72.09	23.27%	2.48	0.10	4.01%
SP10897	Cumin	297.20	94.05	31.65%	2.45	0.15	6.04%
SP10902	Thyme	412.55	64.20	15.56%	2.61	0.07	2.59%
SP10950	Ginseng	76583.90	23140.61	30.22%	4.87	0.13	2.71%
SP10951	Alfalfa	1403.35	246.72	17.58%	3.14	0.08	2.42%
SP10954	Guarana	476.30	174.67	36.67%	2.66	0.12	4.42%
Irradiated							
SP10895	Cinnamon	1045.05	326.05	31.20%	3.00	0.12	4.16%
SP10897	Cumin	42021.90	10853.14	25.83%	4.61	0.11	2.35%
SP10902	Thyme	1353592.50	171668.39	12.68%	6.13	0.06	0.93%
SP10950	Ginseng	1592757.55	389087.65	24.43%	6.19	0.12	1.89%
SP10951	Alfalfa	1666017.85	176542.91	10.60%	6.22	0.05	0.77%
SP10954	Guarana	10740.40	2860.90	26.64%	4.01	0.12	3.11%
Blends							
SP10895	Cinnamon 10%	396.50	138.29	34.88%	2.58	0.14	5.26%
SP10897	Cumin 1%	899.10	749.34	83.34%	2.88	0.22	7.69%
SP10902	Thyme 0.1%	2451.60	3269.40	133.36%	3.18	0.40	12.53%
SP10951	Alfalfa 0.1%	3091.10	1225.07	39.63%	3.47	0.14	4.18%
SP10952	Green Tea 1%	632.05	210.96	33.38%	2.78	0.13	4.73%
SP10954	Guarana 10%	1544.30	684.81	44.34%	3.14	0.21	6.60%

		Windows					
		Linear			Log		
		Mean	SD	CV	Mean	SD	CV
Unirradiated							
SP10895	Cinnamon	368.30	66.87	18.16%	2.56	0.08	3.21%
SP10897	Cumin	372.85	76.82	20.60%	2.56	0.11	4.23%
SP10902	Thyme	583.00	72.71	12.47%	2.76	0.05	1.95%
SP10950	Ginseng	119174.80	32117.79	26.95%	5.06	0.11	2.12%
SP10951	Alfalfa	2218.20	394.61	17.79%	3.34	0.07	2.24%
SP10954	Guarana	612.80	94.15	15.36%	2.78	0.06	2.27%
Irradiated							
SP10895	Cinnamon	1696.75	680.18	40.09%	3.20	0.15	4.67%
SP10897	Cumin	70663.80	40118.08	56.77%	4.78	0.29	6.14%
SP10902	Thyme	2174872.80	301101.69	13.84%	6.33	0.06	0.98%
SP10950	Ginseng	2627969.85	823786.45	31.35%	6.39	0.16	2.56%
SP10951	Alfalfa	2442373.10	425325.26	17.41%	6.38	0.08	1.26%
SP10954	Guarana	22303.75	18047.37	80.92%	4.28	0.21	4.94%
Blends							
SP10895	Cinnamon 10%	485.00	103.58	21.36%	2.68	0.09	3.39%
SP10897	Cumin 1%	21727.35	89153.99	410.33%	3.26	0.61	18.78%
SP10902	Thyme 0.1%	4296.70	5111.43	118.96%	3.38	0.47	13.91%
SP10951	Alfalfa 0.1%	6900.45	6546.80	94.87%	3.74	0.25	6.80%
SP10952	Green Tea 1%	1076.20	776.19	72.12%	2.98	0.19	6.41%
SP10954	Guarana 10%	3094.95	5424.59	175.27%	3.29	0.33	10.16%

Relative sensitivities per product

	Mean	SD
Cinnamon	1.24	0.37
Cumin	1.41	0.65
Thyme	1.43	0.20
Ginseng	1.64	0.48
Alfalfa	1.63	0.40
Guarana	1.38	0.38
Mean	1.46	
Paprika	1.41	

3.2 Participants' Results

35 participants submitted PSL screening results out of 37 sets distributed. In all cases the completed spreadsheets with terminal counts were returned.

3.2.1 The paprika standard

Participants also returned data from the irradiated paprika reference standard. The means, standard deviations and standard errors are tabulated in Table 3.3.

	Mean	SD	SE
SUERC DOS	111583	26522	8387
SUERC WIN	157362	14288	4518
Lab 1	112110	14953	4729
Lab 2	147628	37477	11851
Lab 3	101773	24086	7617
Lab 4	40412	22738	7191
Lab 5	139849	16921	5351
Lab 6	187683	32551	10293
Lab 7	170731	33041	10449
Lab 8	144892	32264	10203
Lab 9	140891	20388	6447
Lab 10	154666	27042	8551
Lab 11	129013	22078	6982
Lab 13	175489	26585	8407
Lab 14	92520	16709	5284
Lab 15	110444	13774	4356
Lab 16	149604	26833	8485
Lab 17	149085	20136	6367
Lab 18	54281	5140	1625
Lab 19	126080	21059	6660
Lab 20	172111	34239	10827
Lab 21	139222	48218	15248
Lab 22	110153	30603	9678
Lab 23	131841	20152	6373
Lab 24	83396	11724	3707
Lab 25	81176	14728	4657
Lab 26	47587	5099	1613
Lab 28	132844	38331	12121
Lab 29	21110	7361	2328
Lab 30	114215	22671	7169
Lab 31	207077	62485	19759
Lab 32	154307	25739	8139
Lab 33	149663	33724	10665
Lab 34	71817	21862	6913
Lab 35	165465	22050	6973
Lab 36	149747	31334	9909
Lab 37	176894	25754	8144

Table 3.3. Participants' paprika standards – mean, standard deviation and standard error from 10 observations

Lab	Round 1	Round 2	Round 3
SUERC Win			1.41
1	0.97	1.25	1.00
2	0.95	1.36	1.32
3		1.22	0.91
4	0.70	0.63	0.36
5			1.25
6		2.29	1.68
7	1.28	1.24	1.53
8	1.17	1.00	1.30
9			1.26
10	1.18	1.34	1.39
11			1.16
13			1.57
14			0.83
15			0.99
16	1.12	1.02	1.34
17	0.65	1.22	1.34
18	0.62		0.49
19	1.02	1.25	1.13
20			1.54
21	1.08	1.16	1.25
22	0.70	0.78	0.99
23	1.06	0.99	1.18
24	0.79	0.92	0.75
25	1.25	1.57	0.73
26	0.72	0.64	0.43
28	1.03	0.81	1.19
29	0.83	1.22	0.19
30			1.02
31			1.86
32	0.81	1.43	1.38
33		1.20	1.34
34			0.64
35		1.26	1.48
36	1.22	1.98	1.34
37			1.59
Mean	0.98	1.21	1.14
SD	0.24	0.37	0.39

Table 3.4 Relativity instrumental sensitivities derived from measurements of the paprika standard (10 each for rounds 1,2 and 3); Round 3 laboratory numbers used

For each of the 3 rounds, sensitivities relative to the SUERC DOS system have been calculated. For each laboratory that participated in Round 3, these sensitivities have been tabulated together with their results from earlier rounds if applicable (Table 3.4).

Overall, Round 3 results appear to be quite similar to those obtained before. The data from round 3 range from 0.19 to 1.86; compared with 0.53 to 1.45 and 0.53 to 1.98. The mean sensitivity for the 3 rounds is very similar, suggesting that comparisons between analyses

conducted in 2005, 2006 and 2007 remains valid. Those laboratories with reduced relative sensitivity may have "dirty" filters.

3.2.2 Screening results

Raw data, comprising the PSL intensities for each test material and participant, are tabulated in Appendix C. These have been represented graphically in Figure 3.3, which shows all the participants' data by product. As in the reference data set, unirradiated samples, fall mostly in the negative and intermediate bands. As expected, all participants have detected positive signals in the ginseng. Irradiated samples again as expected straddle intermediate and positive bands with the exception of the low-sensitivity cinnamon. The blends have a slightly greater spread than the reference data, to be expected from results obtained from a number of different instruments. Overall performance is clearly similar to the reference set.

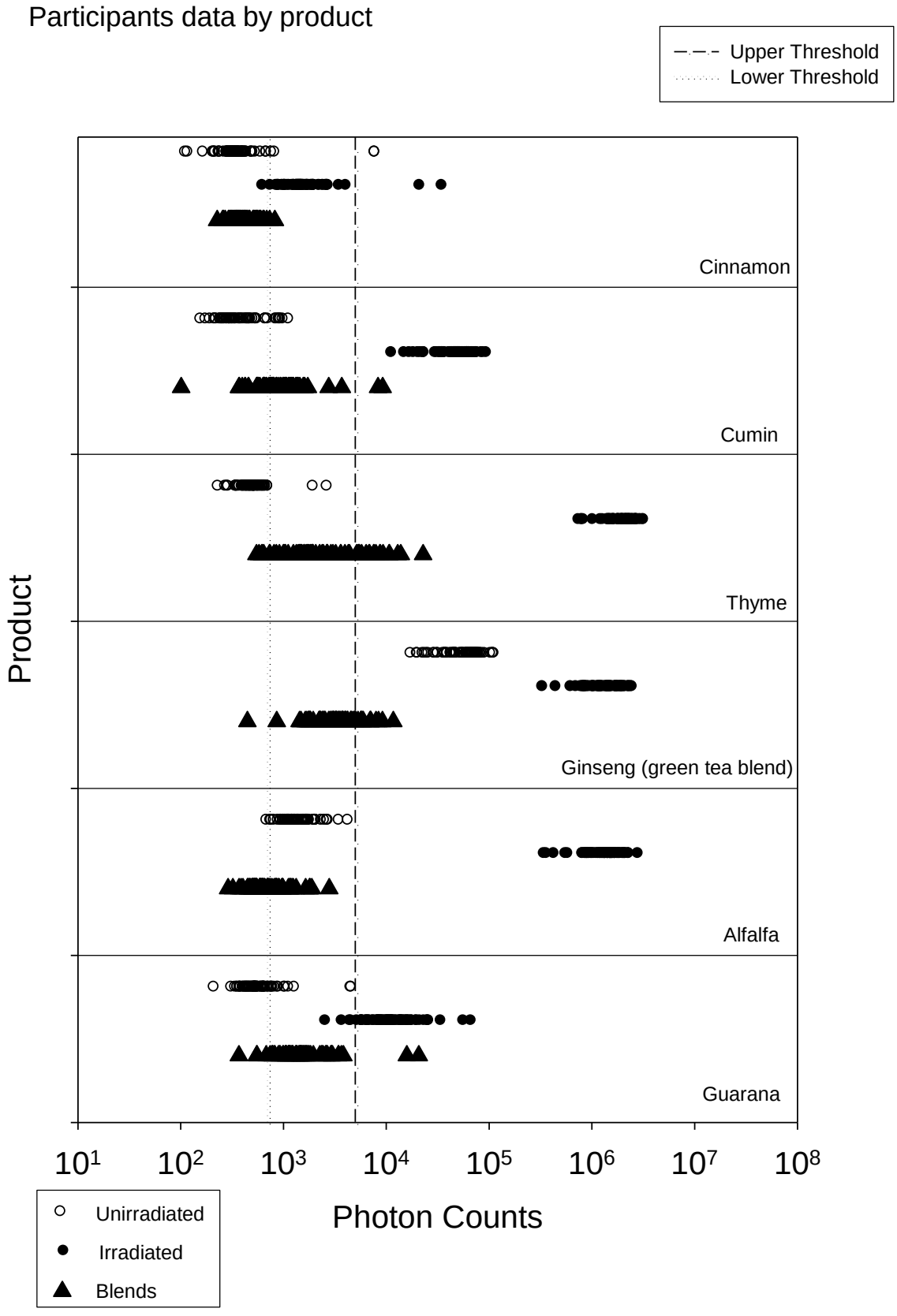


Figure 3.3 Participants' screening data for each product.

Participants' data are also presented as scatter diagrams for each of the categories of sample (irradiated, unirradiated and blended) in Figures 3.4-3.6, arranged by laboratory.

From Figure 3.5 it can be seen that, while every laboratory has positive values for the ginseng sample, there is only one other measurement which exceeds the upper threshold. This suggests a considerable improvement in sample handling in comparison with Round 2, in which some of the Round 1 problems had still not been eliminated.

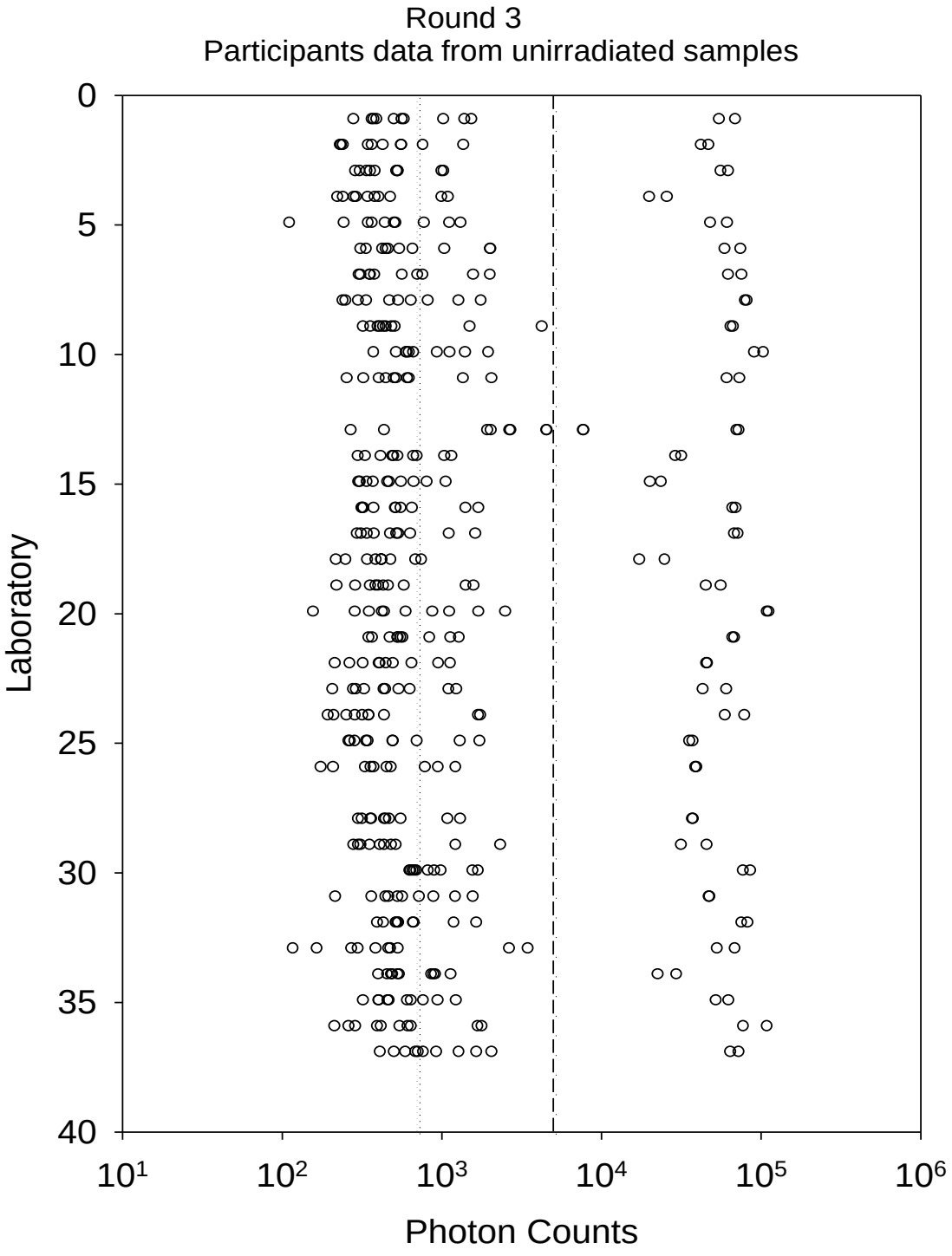


Figure 3.4 Participants' data for unirradiated samples by laboratory

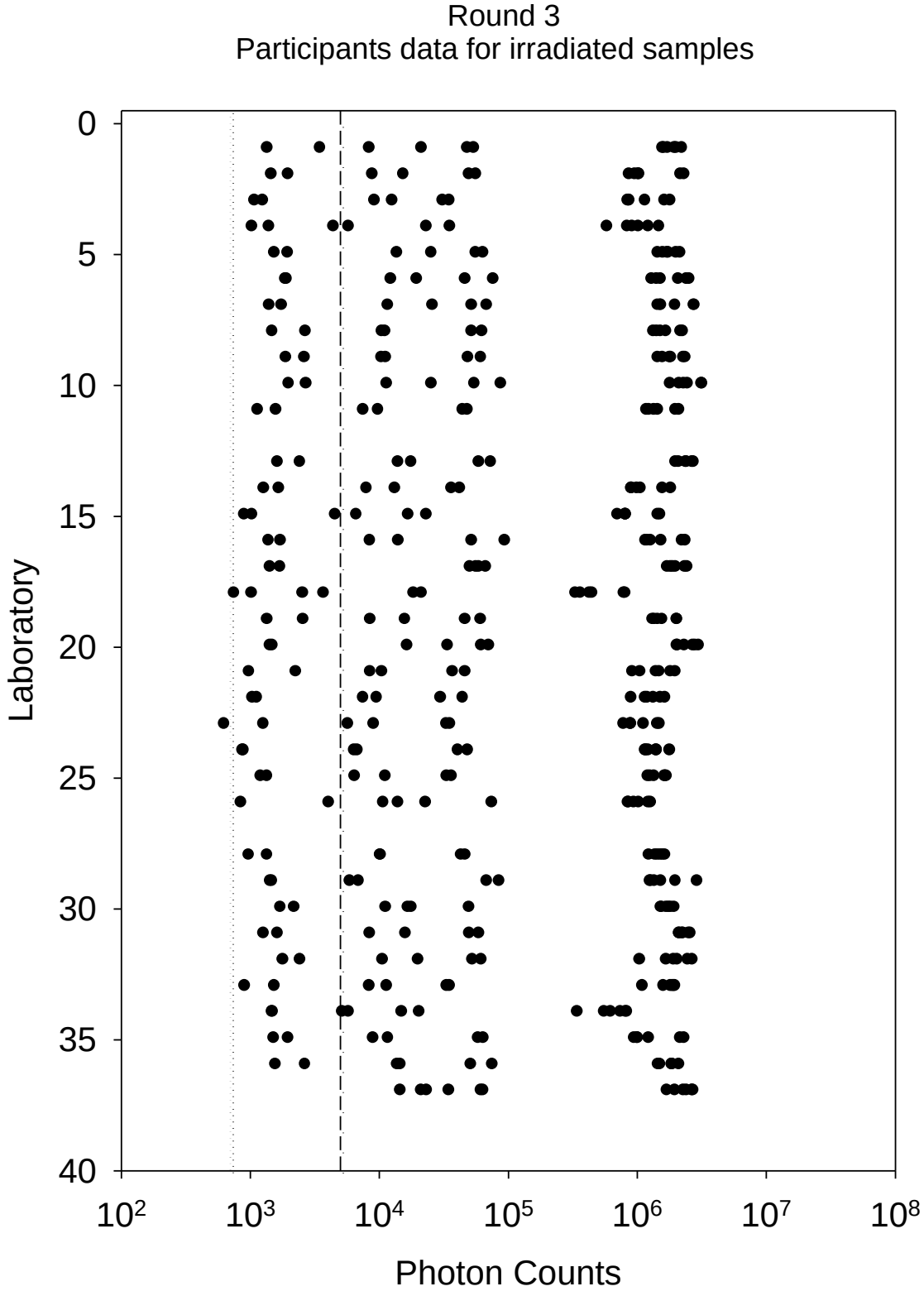


Figure 3.5 Participants' data for irradiated samples by laboratory

The natural range of sample sensitivity is still apparent, as it was in previous rounds, from the observed spread of irradiated results(Figure 3.5) over 4-5 orders of magnitude. Laboratory to laboratory sensitivity variations (eg as seen in the paprika) clearly are very small in comparison with the range of variations from sample to sample.

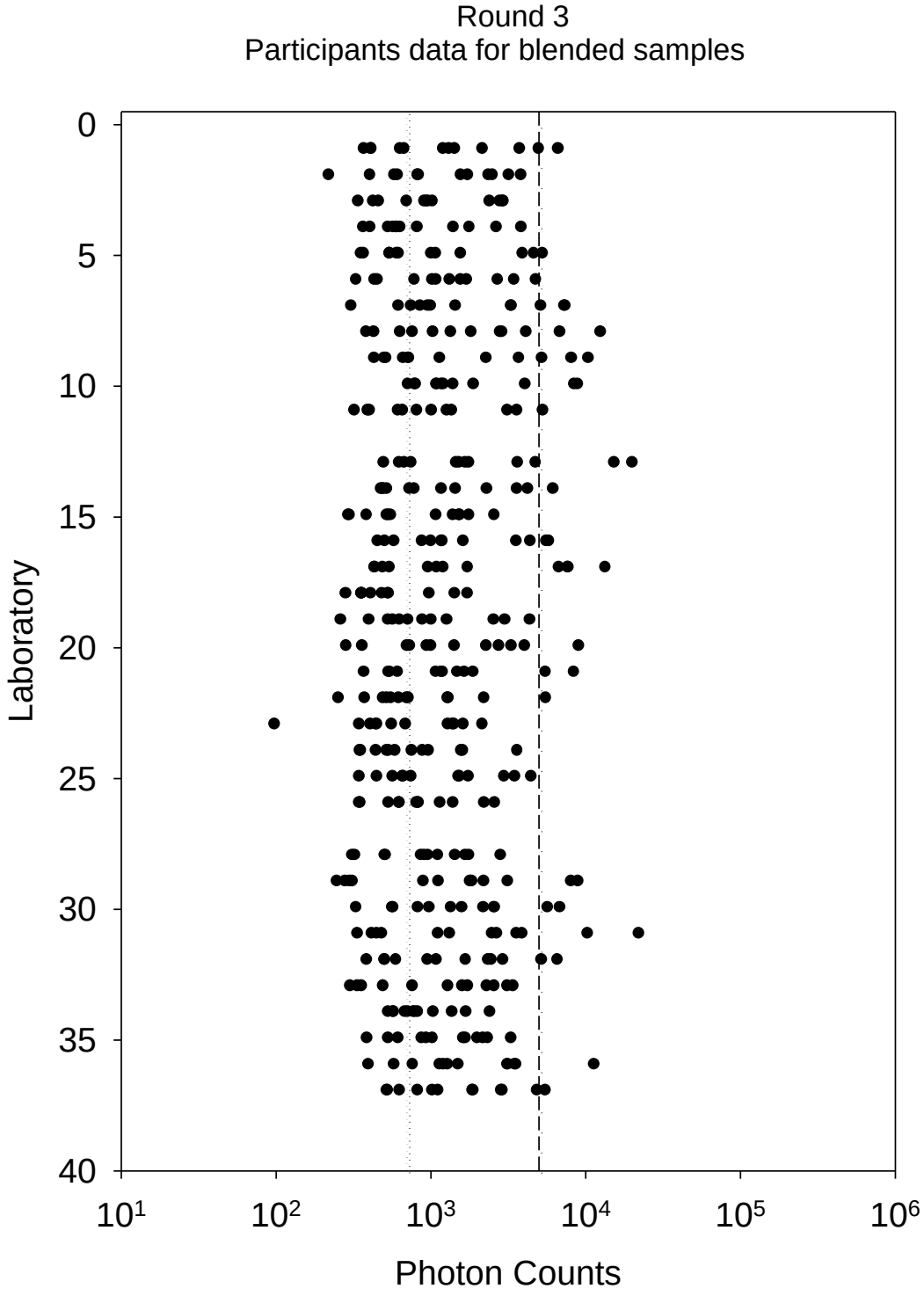


Figure 3.6 Participants' data for blended samples by laboratory and concentration

The results from the blended samples, shown in Figure 3.6, show that far fewer of the blends fall into the positive band than was the case in Round 2. The materials with the lowest sensitivity were blended at the highest concentration, and it is clear that the low sensitivity has reduced the number of positive observations. Conversely, the lowest concentrations used the two most sensitive products (the ginseng has the highest sensitivity as established by the reference analysis, but as explained above this could not be used to make a blend). At each concentration the paring of sensitivity and concentration has led to a similar overall distribution. This again confirms the importance of classifying intermediate results as requiring further attention, as recommended by EN13751.

Figure 3.7 presents all the participants' data as a histogram. Again the anomalous position of the ginseng is readily apparent

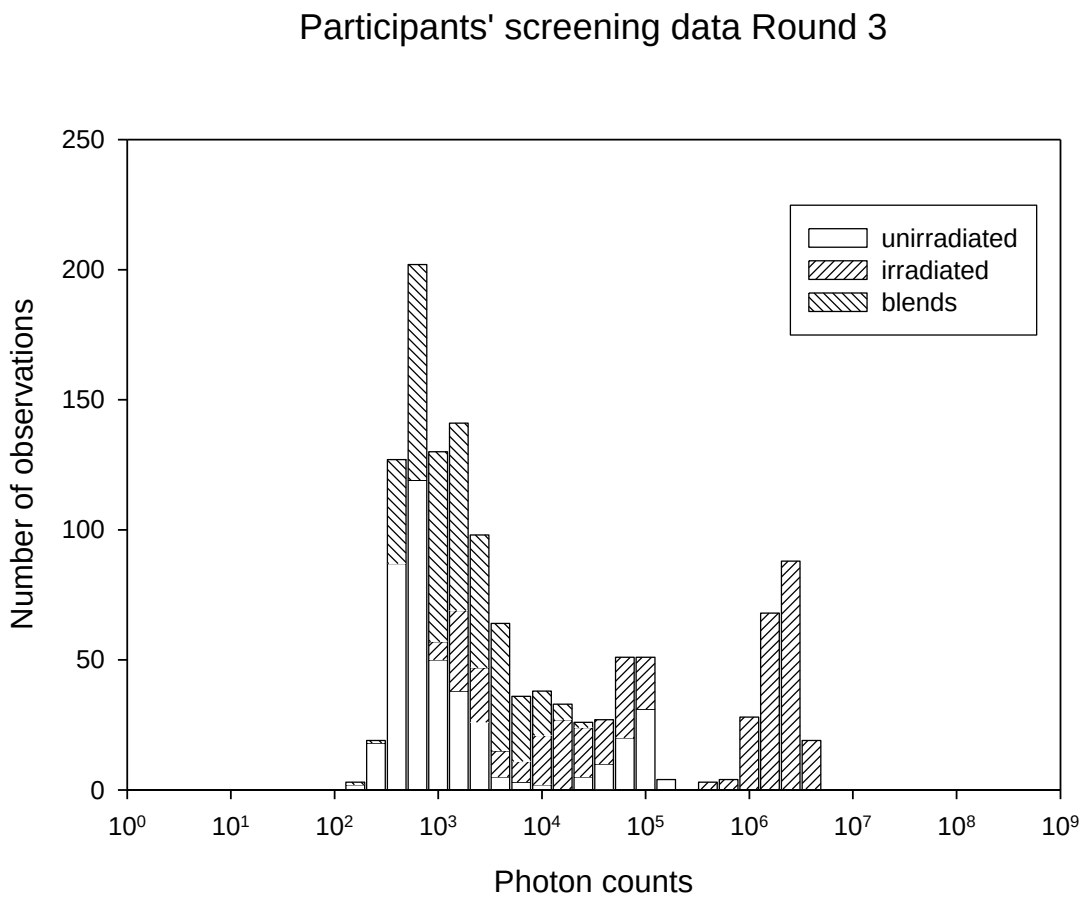


Figure 3.7 Histogram of participants' screening results for all products

3.2.3 Summary Statistics

As described in the Round 1 report (section 2.3), *z*-scores were calculated for the screening measurements. The same calculations were applied in Round 3, using the new reference set to calculate assigned values.

Z-scores are tabulated below to allow each participant to assess their outcomes on a test material by test material basis. Tables 3.5 -3.7 show *z*-scores for each sample by laboratories and tables 3.8-3.10 show *z*-scores by product. Graphical representation, which reveals patterns in the data, follows.

Table 3.5 Round 3 Test Materials: *z*-Scores for Participants 1-13

Description	Status	Lab 1	Lab2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	Lab 9	Lab 10	Lab 11	Lab 13
Cinnamon	U	-0.18	-0.95	0.21	-0.15	-4.21	0.61	0.17	-0.86	0.42	1.08	1.41	14.29
		0.96	-0.83	-0.06	-0.02	2.38	0.26	0.83	0.63	1.48	3.13	0.45	14.27
Cumin	U	1.05	0.86	1.95	-0.60	-0.33	1.55	0.39	0.28	1.34	4.15	1.80	1.38
		0.93	0.69	0.63	-0.37	0.69	2.03	0.96	-0.26	1.12	3.61	1.05	-0.04
Thyme	U	1.55	2.23	-0.64	-0.87	0.72	0.49	3.72	1.15	1.35	2.95	2.94	12.28
		2.49	-3.44	1.80	1.23	-0.49	3.29	2.31	1.96	1.64	3.38	0.82	10.24
Ginseng	U	-0.09	-1.71	-0.42	-3.33	-0.48	0.17	-0.42	0.37	-0.31	0.81	0.12	0.08
		-0.85	-1.35	-0.78	-4.16	-1.28	-0.58	0.22	0.46	-0.19	1.25	-0.48	-0.03
Alfalfa	U	0.80	0.14	-1.50	-1.68	-1.04	2.38	2.33	1.58	0.67	2.18	2.48	2.39
		0.23	-3.22	-1.66	-1.14	-0.10	2.35	0.94	-0.28	6.61	0.29	0.10	4.02
Guarana	U	3.11	-0.11	0.69	-0.55	2.07	0.06	-0.79	1.38	0.05	1.16	0.60	8.63
		0.89	0.88	-0.54	-0.34	0.56	3.16	2.00	2.29	-0.78	0.60	1.18	8.60
Cinnamon	I	1.16	1.42	0.89	1.27	1.61	2.37	2.04	1.47	3.47	3.57	0.57	3.19
		4.43	2.46	0.38	0.21	2.44	2.28	1.30	3.54	2.32	2.49	1.71	1.80
Cumin	I	1.28	0.92	-0.95	-0.44	1.94	2.66	2.21	1.13	1.77	3.20	0.80	2.48
		0.81	1.42	-0.50	-2.13	1.41	0.66	1.13	1.85	0.86	1.31	0.48	1.65
Thyme	I	4.12	4.40	2.49	0.98	3.84	4.74	5.73	4.22	4.31	6.78	3.23	5.50
		3.36	3.93	1.75	-1.84	3.35	5.11	5.81	3.95	4.55	6.86	3.71	5.69
Ginseng	I	0.70	-3.98	-5.78	-2.48	0.62	2.36	-0.51	-1.68	1.19	3.91	-2.74	1.95
		1.78	-5.59	-5.58	-5.89	0.77	-1.12	1.87	-0.47	-0.93	3.30	-2.38	3.72
Alfalfa	I	0.20	-1.64	-0.96	-1.82	-0.11	0.06	0.09	0.43	0.21	1.31	-0.11	1.26
		0.30	-1.40	-2.03	-3.50	0.23	-0.52	-0.12	-0.18	0.67	0.70	-0.36	1.75
Guarana	I	-0.62	-0.42	-0.30	-1.90	3.23	0.72	3.31	0.16	0.41	0.46	-0.07	1.98
		2.64	1.50	0.81	-2.84	1.10	2.33	0.53	0.37	0.14	3.25	-1.00	1.16
Cinnamon	B	0.04	0.32	0.48	0.01	0.02	0.68	1.69	0.15	1.91	2.50	0.30	1.72
		0.39	-1.64	-0.24	0.34	-0.11	-0.34	-0.57	0.51	1.00	2.13	-0.42	0.98
Cumin	B	-0.29	0.21	0.41	-0.50	-0.61	1.46	0.59	0.06	0.86	1.24	-0.23	1.60
		-1.15	0.24	-0.11	-0.65	-0.61	1.15	0.02	1.18	-0.06	0.76	-0.36	1.42
Thyme	B	1.66	0.53	0.71	-0.98	-0.33	0.17	1.77	2.35	1.39	1.92	-0.40	0.20
		1.34	1.05	0.76	-0.96	1.40	-0.68	1.76	1.13	2.14	-0.20	1.41	0.00
Alfalfa	B	0.85	0.36	0.12	0.93	1.48	1.57	0.47	0.05	-5.64	3.43	0.30	0.76
		-0.81	-1.46	-0.49	-0.18	0.98	0.59	1.79	2.65	3.17	1.10	0.72	1.55
Green tea	B	0.47	-0.02	1.57	1.11	0.18	-1.00	1.62	0.27	0.69	0.98	-1.33	0.81
		2.39	0.14	-0.79	0.28	0.11	5.06	1.26	3.77	-0.43	2.30	1.09	0.47
Guarana	B	-0.06	1.29	-0.58	0.57	-0.61	-0.47	1.88	-0.56	2.11	-0.44	-0.13	5.64
		0.12	0.30	-0.71	0.08	0.30	-0.59	0.14	1.53	1.10	0.70	0.02	5.08

Table 3.6 Round 3 Test Materials: *z*-Scores for Participants 14-25

Description	Status	Lab 14	Lab 15	Lab 16	Lab 17	Lab 18	Lab 19	Lab 20	Lab 21	Lab 22	Lab 23	Lab 24	Lab 25
Cinnamon	U	0.10	1.03	0.34	0.05	1.22	1.22	0.80	0.77	-0.43	-1.51	-0.09	0.71
		0.55	0.23	0.38	0.66	-1.29	0.87	1.63	1.00	0.41	0.49	0.37	0.61
Cumin	U	2.77	0.29	2.08	0.95	1.65	-0.63	-1.63	1.61	-0.70	1.43	-0.22	-0.07
		2.62	1.51	0.50	0.40	-0.25	0.16	0.14	3.29	1.46	0.06	-1.01	-0.13
Thyme	U	1.42	3.41	1.74	1.80	-0.91	0.10	0.65	2.37	1.47	-1.97	-0.78	3.71
		0.33	1.13	1.67	1.97	0.38	1.03	2.65	2.17	0.23	0.60	-0.77	-2.08
Ginseng	U	-2.92	-3.60	-0.23	0.04	-3.44	-1.47	1.41	-0.22	-1.46	-0.50	-0.56	-2.10
		-2.64	-4.13	-0.07	-0.14	-4.63	-0.77	1.50	-0.13	-1.41	-1.62	0.34	-2.26
Alfalfa	U	-0.87	-1.33	0.31	-1.04	-3.34	0.32	1.37	-0.22	-0.95	-1.10	1.52	1.49
		-1.44	-2.87	1.37	1.12	-3.82	0.97	3.59	-0.94	-1.94	-0.46	1.35	-0.17
Guarana	U	0.46	-0.97	-0.60	1.35	-0.20	-0.09	2.53	0.69	-0.33	0.73	-0.04	0.44
		0.67	0.86	1.44	0.27	-0.19	1.01	3.44	0.67	1.42	1.33	-2.73	0.41
Cinnamon	I	1.88	-0.26	1.24	1.96	-0.90	1.18	1.33	0.03	0.25	0.93	-0.32	1.16
		0.96	0.22	2.00	1.33	0.19	3.39	1.49	2.93	0.52	-1.51	-0.36	0.77
Cumin	I	0.28	-3.42	3.50	1.63	-2.45	0.66	2.34	0.66	0.48	-0.43	0.83	-0.65
		-0.32	-2.12	1.13	1.00	-3.04	1.76	1.82	-0.24	-1.11	-0.69	0.14	-0.31
Thyme	I	2.60	0.81	4.59	4.48	-3.66	3.43	5.64	3.20	1.79	0.78	0.63	1.77
		1.48	1.12	4.11	4.84	-3.79	3.41	6.40	2.56	1.17	1.04	2.46	2.00
Ginseng	I	-3.73	-6.17	-2.66	0.65	-11.63	-0.93	3.37	-1.22	-5.27	-6.51	-1.16	0.29
		-5.18	-7.46	-2.93	1.15	-14.31	-0.25	2.30	-0.70	-2.97	-3.28	-2.81	-2.52
Alfalfa	I	-1.50	-2.23	0.11	1.06	-5.27	-0.34	2.37	-1.29	-0.42	-1.90	-0.75	-0.36
		-1.86	-2.27	-0.59	0.89	-4.64	-0.46	1.16	-1.80	-0.81	-1.93	-0.95	-0.63
Guarana	I	0.98	-2.74	-0.58	6.01	-4.75	1.60	4.25	-0.56	-0.16	-1.95	-1.55	0.38
		-0.80	-1.43	1.19	6.61	-3.45	-0.56	1.74	0.17	-1.00	-0.34	-1.35	-1.54
Cinnamon	B	0.97	-0.71	0.69	1.25	-0.07	0.29	-0.04	1.27	-1.18	-0.19	-0.12	-0.20
		0.93	-0.66	1.46	0.54	-0.82	-1.06	-0.81	0.04	1.13	0.35	-0.16	0.66
Cumin	B	-0.03	-1.28	0.90	0.52	-1.15	1.07	-0.02	0.90	-0.57	-1.00	-1.01	1.40
		0.11	-0.58	3.09	0.96	-1.41	-0.32	0.60	-0.63	-0.11	-3.96	-0.63	-0.21
Thyme	B	1.57	0.06	1.47	1.81	-1.11	-0.78	1.99	1.91	-0.93	-0.06	0.10	0.95
		1.16	0.62	-0.41	2.42	-0.44	-0.55	-0.03	0.13	-0.13	-1.04	-0.45	0.05
Alfalfa	B	0.73	-2.13	2.15	3.01	-2.04	1.31	1.08	-1.22	-0.74	-1.67	0.73	0.16
		-0.61	-1.42	1.31	2.60	-1.48	0.20	-0.08	2.00	2.01	-0.82	-1.75	1.37
Green tea	B	-0.39	-0.39	1.34	-0.59	-1.63	-0.32	1.58	2.03	-1.48	-0.87	0.01	3.63
		-0.68	-0.32	-0.49	-0.97	-0.63	-0.10	0.61	0.14	-0.58	0.54	-0.39	-0.10
Guarana	B	0.14	0.24	-0.27	-0.44	-2.79	1.33	1.88	0.19	-1.33	0.09	-0.88	-1.51
		-0.30	-0.47	0.38	0.51	-1.95	-0.61	1.10	-0.27	-0.11	-0.09	-1.22	-1.24

Table 3.7 Round 3 Test Materials: *z*-Scores for Participants 26-37

Description	Status	Lab 26	Lab 28	Lab 29	Lab 30	Lab 31	Lab 32	Lab 33	Lab 34	Lab 35	Lab 36	Lab 37
Cinnamon	U	0.91	0.94	1.75	4.50	0.94	1.70	-3.99	2.26	1.41	1.55	4.20
		-1.47	0.89	2.20	3.66	-1.33	1.31	-2.48	2.60	1.38	-0.06	3.72
Cumin	U	0.56	0.44	0.08	3.77	1.56	1.97	1.02	3.37	1.58	-0.12	3.59
		-1.30	0.28	0.28	3.49	1.43	2.64	0.27	3.55	0.49	-0.72	1.20
Thyme	U	-0.32	0.66	-0.70	3.01	1.90	1.72	1.23	0.93	0.99	0.02	2.62
		1.28	0.80	0.25	3.09	2.33	1.80	-2.37	0.10	2.77	3.13	1.56
Ginseng	U	-1.93	-2.10	-2.66	0.29	-1.34	0.21	-0.11	-3.75	-1.00	1.41	0.08
		-1.98	-2.12	-1.44	0.64	-1.30	0.50	-0.95	-2.89	-0.41	0.29	-0.32
Alfalfa	U	-0.52	-1.16	-0.52	1.34	-0.55	-0.67	3.92	-0.90	-1.98	1.31	2.46
		-1.96	-0.13	3.20	0.89	0.92	1.22	5.45	-2.33	-0.47	1.63	1.21
Guarana	U	2.13	0.84	0.58	1.48	2.59	1.49	0.69	0.33	2.03	0.79	3.93
		0.09	0.23	-1.29	1.67	1.81	0.71	0.18	0.75	1.38	1.21	1.76
Cinnamon	I	4.98	1.15	1.45	2.85	1.80	3.20	1.61	1.51	1.57	3.50	10.71
		-0.46	0.01	1.35	1.98	0.93	2.14	-0.23	1.45	2.46	1.67	12.43
Cumin	I	2.57	0.67	3.08	-5.03	0.95	1.82	-0.66	-3.88	1.96	2.60	1.94
		-2.19	0.36	2.21	0.93	1.65	1.16	-0.46	-2.65	1.60	1.04	1.77
Thyme	I	-0.48	1.53	3.21	3.08	5.27	5.55	2.82	-4.28	3.89	2.78	5.62
		-0.13	1.80	6.18	2.59	5.04	4.93	3.16	-3.40	4.42	3.71	5.51
Ginseng	I	-4.04	-2.38	-2.04	0.84	2.55	-3.86	1.14	-8.57	-4.20	-0.86	3.74
		-5.76	-1.40	-2.24	-0.29	3.08	1.63	1.66	-6.15	-4.76	-0.59	1.82
Alfalfa	I	-1.70	-0.12	-0.34	0.46	1.31	1.18	0.26	-5.46	-0.73	0.83	0.49
		-2.05	0.07	0.09	0.09	1.56	0.44	-1.12	-3.66	-1.46	0.81	1.58
Guarana	I	1.17	0.08	-1.29	1.79	1.63	0.21	-0.61	-2.28	-0.38	1.13	2.95
		0.24	0.07	-1.82	1.99	-0.60	2.42	0.47	-1.91	0.55	1.32	1.32
Cinnamon	B	-0.20	-0.39	-0.64	1.39	0.42	0.18	-0.28	1.43	0.19	0.26	1.13
		-0.13	-0.52	-1.24	-0.34	-0.28	1.01	-0.61	1.20	1.66	1.46	1.17
Cumin	B	-0.33	0.80	4.68	0.22	1.15	0.76	-1.42	0.09	0.33	0.07	0.63
		-0.33	0.40	4.89	0.55	2.51	0.50	1.10	0.67	0.64	0.96	-0.32
Thyme	B	0.63	0.15	0.23	0.61	2.96	1.64	0.50	-0.78	0.44	0.95	0.28
		-0.05	-0.02	0.25	-0.08	2.14	1.38	0.62	-0.62	0.34	0.83	0.26
Alfalfa	B	-0.74	-1.41	-0.75	2.10	0.71	-0.41	0.29	-1.55	0.48	0.32	1.63
		-3.67	0.00	0.32	2.64	0.95	0.11	0.56	-0.47	-0.58	4.17	1.99
Green tea	B	1.06	-0.46	-2.43	-0.08	-0.64	0.06	-0.56	0.50	1.55	2.60	1.13
		1.17	-0.48	-2.09	3.31	-0.87	-0.49	3.60	-0.06	-0.31	2.21	2.13
Guarana	B	-1.94	-0.94	-0.40	1.01	1.28	1.16	-1.20	-1.11	0.38	0.23	1.60
		-0.34	-0.72	-0.86	1.36	-0.40	0.45	0.34	0.04	0.44	2.02	1.56

Figures 3.8-3.11 show the *z*-scores first by sample and then by laboratory.

In figure 3.8 it appears the spread of *z*-scores is similar for the irradiated and unirradiated materials, and that the grouping is tighter than in previous rounds. This may be a result of better sample handling or merely a consequence of the much lower number of samples or even of the inherent variability of the products. For the blends results are similar to Round 2, with a narrower range of *z*-scores than the other categories. No product is distinctly more or less dispersed than any of the others.

When analysed by laboratory in figures 3.9-2.11, it is again apparent that the *z*-scores are more tightly grouped around zero than was the case in previous rounds. For unirradiated materials (Figure 3.9) one laboratory has outlying positive scores, suggestive of a contamination problem in 4 aliquots (2 samples). There is a slight bias towards positive scores but the spread is almost entirely confined between ± 5. The ginseng will not appear as elevated z-scores since the reference data are also positive.

For irradiated materials (Figure 3.10) most of the scores lie between ± 10. Outliers occur in pairs, deriving from the 2 aliquots of a single sample in each case.

For blends (Figure 3.11) most scores lie between ± 5 like the unirradiated samples, but the extreme values here do not always occur in pairs. This may imply that spread is associated with inhomogeneity in the blend.

There is of course no guarantee of homogeneity in the other two categories, although it is clearly the blends which have the highest coefficients of variance as shown in the summary statistics in Table 3.2.

Table 3.8 Round 3 Test Materials: *z*-Scores by product for Participants 1-13

Description	Cinnamon	Cumin	Thyme	Ginseng	Alfalfa	Guarana	Cinnamon	Cumin	Thyme	Ginseng	Alfalfa	Guarana	Cinnamon	Cumin	Thyme	Alfalfa	Green tea	Guarana
Status	U	U	U	U	U	U	I	I	I	I	I	I	B	B	B	B	B	B
Lab 1	-0.18	1.05	1.55	-0.09	0.80	3.11	1.16	1.28	4.12	0.70	0.20	-0.62	0.04	-0.29	1.66	0.85	0.47	-0.06
	0.96	0.93	2.49	-0.85	0.23	0.89	4.43	0.81	3.36	1.78	0.30	2.64	0.39	-1.15	1.34	-0.81	2.39	0.12
Lab2	-0.95	0.86	2.23	-1.71	0.14	-0.11	1.42	0.92	4.40	-3.98	-1.64	-0.42	0.32	0.21	0.53	0.36	-0.02	1.29
	-0.83	0.69	-3.44	-1.35	-3.22	0.88	2.46	1.42	3.93	-5.59	-1.40	1.50	-1.64	0.24	1.05	-1.46	0.14	0.30
Lab 3	0.21	1.95	-0.64	-0.42	-1.50	0.69	0.89	-0.95	2.49	-5.78	-0.96	-0.30	0.48	0.41	0.71	0.12	1.57	-0.58
	-0.06	0.63	1.80	-0.78	-1.66	-0.54	0.38	-0.50	1.75	-5.58	-2.03	0.81	-0.24	-0.11	0.76	-0.49	-0.79	-0.71
Lab 4	-0.15	-0.60	-0.87	-3.33	-1.68	-0.55	1.27	-0.44	0.98	-2.48	-1.82	-1.90	0.01	-0.50	-0.98	0.93	1.11	0.57
	-0.02	-0.37	1.23	-4.16	-1.14	-0.34	0.21	-2.13	-1.84	-5.89	-3.50	-2.84	0.34	-0.65	-0.96	-0.18	0.28	0.08
Lab 5	-4.21	-0.33	0.72	-0.48	-1.04	2.07	1.61	1.94	3.84	0.62	-0.11	3.23	0.02	-0.61	-0.33	1.48	0.18	-0.61
	2.38	0.69	-0.49	-1.28	-0.10	0.56	2.44	1.41	3.35	0.77	0.23	1.10	-0.11	-0.61	1.40	0.98	0.11	0.30
Lab 6	0.61	1.55	0.49	0.17	2.38	0.06	2.37	2.66	4.74	2.36	0.06	0.72	0.68	1.46	0.17	1.57	-1.00	-0.47
	0.26	2.03	3.29	-0.58	2.35	3.16	2.28	0.66	5.11	-1.12	-0.52	2.33	-0.34	1.15	-0.68	0.59	5.06	-0.59
Lab 7	0.17	0.39	3.72	-0.42	2.33	-0.79	2.04	2.21	5.73	-0.51	0.09	3.31	1.69	0.59	1.77	0.47	1.62	1.88
	0.83	0.96	2.31	0.22	0.94	2.00	1.30	1.13	5.81	1.87	-0.12	0.53	-0.57	0.02	1.76	1.79	1.26	0.14
Lab 8	-0.86	0.28	1.15	0.37	1.58	1.38	1.47	1.13	4.22	-1.68	0.43	0.16	0.15	0.06	2.35	0.05	0.27	-0.56
	0.63	-0.26	1.96	0.46	-0.28	2.29	3.54	1.85	3.95	-0.47	-0.18	0.37	0.51	1.18	1.13	2.65	3.77	1.53
Lab 9	0.42	1.34	1.35	-0.31	0.67	0.05	3.47	1.77	4.31	1.19	0.21	0.41	1.91	0.86	1.39	-5.64	0.69	2.11
	1.48	1.12	1.64	-0.19	6.61	-0.78	2.32	0.86	4.55	-0.93	0.67	0.14	1.00	-0.06	2.14	3.17	-0.43	1.10
Lab 10	1.08	4.15	2.95	0.81	2.18	1.16	3.57	3.20	6.78	3.91	1.31	0.46	2.50	1.24	1.92	3.43	0.98	-0.44
	3.13	3.61	3.38	1.25	0.29	0.60	2.49	1.31	6.86	3.30	0.70	3.25	2.13	0.76	-0.20	1.10	2.30	0.70
Lab 11	1.41	1.80	2.94	0.12	2.48	0.60	0.57	0.80	3.23	-2.74	-0.11	-0.07	0.30	-0.23	-0.40	0.30	-1.33	-0.13
	0.45	1.05	0.82	-0.48	0.10	1.18	1.71	0.48	3.71	-2.38	-0.36	-1.00	-0.42	-0.36	1.41	0.72	1.09	0.02
Lab 13	14.29	1.38	12.28	0.08	2.39	8.63	3.19	2.48	5.50	1.95	1.26	1.98	1.72	1.60	0.20	0.76	0.81	5.64
	14.27	-0.04	10.24	-0.03	4.02	8.60	1.80	1.65	5.69	3.72	1.75	1.16	0.98	1.42	0.00	1.55	0.47	5.08

Table 3.9 Round 3 Test Materials: *z*-Scores by product for Participants 14-25

Description	Cinnamon	Cumin	Thyme	Ginseng	Alfalfa	Guarana	Cinnamon	Cumin	Thyme	Ginseng	Alfalfa	Guarana	Cinnamon	Cumin	Thyme	Alfalfa	Green tea	Guarana
Status	U	U	U	U	U	U	I	I	I	I	I	I	B	B	B	B	B	B
Lab 14	0.10	2.77	1.42	-2.92	-0.87	0.46	1.88	0.28	2.60	-3.73	-1.50	0.98	0.97	-0.03	1.57	0.73	-0.39	0.14
	0.55	2.62	0.33	-2.64	-1.44	0.67	0.96	-0.32	1.48	-5.18	-1.86	-0.80	0.93	0.11	1.16	-0.61	-0.68	-0.30
Lab 15	1.03	0.29	3.41	-3.60	-1.33	-0.97	-0.26	-3.42	0.81	-6.17	-2.23	-2.74	-0.71	-1.28	0.06	-2.13	-0.39	0.24
	0.23	1.51	1.13	-4.13	-2.87	0.86	0.22	-2.12	1.12	-7.46	-2.27	-1.43	-0.66	-0.58	0.62	-1.42	-0.32	-0.47
Lab 16	0.34	2.08	1.74	-0.23	0.31	-0.60	1.24	3.50	4.59	-2.66	0.11	-0.58	0.69	0.90	1.47	2.15	1.34	-0.27
	0.38	0.50	1.67	-0.07	1.37	1.44	2.00	1.13	4.11	-2.93	-0.59	1.19	1.46	3.09	-0.41	1.31	-0.49	0.38
Lab 17	0.05	0.95	1.80	0.04	-1.04	1.35	1.96	1.63	4.48	0.65	1.06	6.01	1.25	0.52	1.81	3.01	-0.59	-0.44
	0.66	0.40	1.97	-0.14	1.12	0.27	1.33	1.00	4.84	1.15	0.89	6.61	0.54	0.96	2.42	2.60	-0.97	0.51
Lab 18	1.22	1.65	-0.91	-3.44	-3.34	-0.20	-0.90	-2.45	-3.66	-11.63	-5.27	-4.75	-0.07	-1.15	-1.11	-2.04	-1.63	-2.79
	-1.29	-0.25	0.38	-4.63	-3.82	-0.19	0.19	-3.04	-3.79	-14.31	-4.64	-3.45	-0.82	-1.41	-0.44	-1.48	-0.63	-1.95
Lab 19	1.22	-0.63	0.10	-1.47	0.32	-0.09	1.18	0.66	3.43	-0.93	-0.34	1.60	0.29	1.07	-0.78	1.31	-0.32	1.33
	0.87	0.16	1.03	-0.77	0.97	1.01	3.39	1.76	3.41	-0.25	-0.46	-0.56	-1.06	-0.32	-0.55	0.20	-0.10	-0.61
Lab 20	0.80	-1.63	0.65	1.41	1.37	2.53	1.33	2.34	5.64	3.37	2.37	4.25	-0.04	-0.02	1.99	1.08	1.58	1.88
	1.63	0.14	2.65	1.50	3.59	3.44	1.49	1.82	6.40	2.30	1.16	1.74	-0.81	0.60	-0.03	-0.08	0.61	1.10
Lab 21	0.77	1.61	2.37	-0.22	-0.22	0.69	0.03	0.66	3.20	-1.22	-1.29	-0.56	1.27	0.90	1.91	-1.22	2.03	0.19
	1.00	3.29	2.17	-0.13	-0.94	0.67	2.93	-0.24	2.56	-0.70	-1.80	0.17	0.04	-0.63	0.13	2.00	0.14	-0.27
Lab 22	-0.43	-0.70	1.47	-1.46	-0.95	-0.33	0.25	0.48	1.79	-5.27	-0.42	-0.16	-1.18	-0.57	-0.93	-0.74	-1.48	-1.33
	0.41	1.46	0.23	-1.41	-1.94	1.42	0.52	-1.11	1.17	-2.97	-0.81	-1.00	1.13	-0.11	-0.13	2.01	-0.58	-0.11
Lab 23	-1.51	1.43	-1.97	-0.50	-1.10	0.73	0.93	-0.43	0.78	-6.51	-1.90	-1.95	-0.19	-1.00	-0.06	-1.67	-0.87	0.09
	0.49	0.06	0.60	-1.62	-0.46	1.33	-1.51	-0.69	1.04	-3.28	-1.93	-0.34	0.35	-3.96	-1.04	-0.82	0.54	-0.09
Lab 24	-0.09	-0.22	-0.78	-0.56	1.52	-0.04	-0.32	0.83	0.63	-1.16	-0.75	-1.55	-0.12	-1.01	0.10	0.73	0.01	-0.88
	0.37	-1.01	-0.77	0.34	1.35	-2.73	-0.36	0.14	2.46	-2.81	-0.95	-1.35	-0.16	-0.63	-0.45	-1.75	-0.39	-1.22
Lab 25	0.71	-0.07	3.71	-2.10	1.49	0.44	1.16	-0.65	1.77	0.29	-0.36	0.38	-0.20	1.40	0.95	0.16	3.63	-1.51
	0.61	-0.13	-2.08	-2.26	-0.17	0.41	0.77	-0.31	2.00	-2.52	-0.63	-1.54	0.66	-0.21	0.05	1.37	-0.10	-1.24

Table 3.10 Round 3 Test Materials: *z*-Scores by product for Participants 26-37

Description	Cinnamon	Cumin	Thyme	Ginseng	Alfalfa	Guarana	Cinnamon	Cumin	Thyme	Ginseng	Alfalfa	Guarana	Cinnamon	Cumin	Thyme	Alfalfa	Green tea	Guarana
Status	U	U	U	U	U	U	I	I	I	I	I	I	B	B	B	B	B	B
Lab 26	0.91	0.56	-0.32	-1.93	-0.52	2.13	4.98	2.57	-0.48	-4.04	-1.70	1.17	-0.20	-0.33	0.63	-0.74	1.06	-1.94
	-1.47	-1.30	1.28	-1.98	-1.96	0.09	-0.46	-2.19	-0.13	-5.76	-2.05	0.24	-0.13	-0.33	-0.05	-3.67	1.17	-0.34
Lab 28	0.94	0.44	0.66	-2.10	-1.16	0.84	1.15	0.67	1.53	-2.38	-0.12	0.08	-0.39	0.80	0.15	-1.41	-0.46	-0.94
	0.89	0.28	0.80	-2.12	-0.13	0.23	0.01	0.36	1.80	-1.40	0.07	0.07	-0.52	0.40	-0.02	0.00	-0.48	-0.72
Lab 29	1.75	0.08	-0.70	-2.66	-0.52	0.58	1.45	3.08	3.21	-2.04	-0.34	-1.29	-0.64	4.68	0.23	-0.75	-2.43	-0.40
	2.20	0.28	0.25	-1.44	3.20	-1.29	1.35	2.21	6.18	-2.24	0.09	-1.82	-1.24	4.89	0.25	0.32	-2.09	-0.86
Lab 30	4.50	3.77	3.01	0.29	1.34	1.48	2.85	-5.03	3.08	0.84	0.46	1.79	1.39	0.22	0.61	2.10	-0.08	1.01
	3.66	3.49	3.09	0.64	0.89	1.67	1.98	0.93	2.59	-0.29	0.09	1.99	-0.34	0.55	-0.08	2.64	3.31	1.36
Lab 31	0.94	1.56	1.90	-1.34	-0.55	2.59	1.80	0.95	5.27	2.55	1.31	1.63	0.42	1.15	2.96	0.71	-0.64	1.28
	-1.33	1.43	2.33	-1.30	0.92	1.81	0.93	1.65	5.04	3.08	1.56	-0.60	-0.28	2.51	2.14	0.95	-0.87	-0.40
Lab 32	1.70	1.97	1.72	0.21	-0.67	1.49	3.20	1.82	5.55	-3.86	1.18	0.21	0.18	0.76	1.64	-0.41	0.06	1.16
	1.31	2.64	1.80	0.50	1.22	0.71	2.14	1.16	4.93	1.63	0.44	2.42	1.01	0.50	1.38	0.11	-0.49	0.45
Lab 33	-3.99	1.02	1.23	-0.11	3.92	0.69	1.61	-0.66	2.82	1.14	0.26	-0.61	-0.28	-1.42	0.50	0.29	-0.56	-1.20
	-2.48	0.27	-2.37	-0.95	5.45	0.18	-0.23	-0.46	3.16	1.66	-1.12	0.47	-0.61	1.10	0.62	0.56	3.60	0.34
Lab 34	2.26	3.37	0.93	-3.75	-0.90	0.33	1.51	-3.88	-4.28	-8.57	-5.46	-2.28	1.43	0.09	-0.78	-1.55	0.50	-1.11
	2.60	3.55	0.10	-2.89	-2.33	0.75	1.45	-2.65	-3.40	-6.15	-3.66	-1.91	1.20	0.67	-0.62	-0.47	-0.06	0.04
Lab 35	1.41	1.58	0.99	-1.00	-1.98	2.03	1.57	1.96	3.89	-4.20	-0.73	-0.38	0.19	0.33	0.44	0.48	1.55	0.38
	1.38	0.49	2.77	-0.41	-0.47	1.38	2.46	1.60	4.42	-4.76	-1.46	0.55	1.66	0.64	0.34	-0.58	-0.31	0.44
Lab 36	1.55	-0.12	0.02	1.41	1.31	0.79	3.50	2.60	2.78	-0.86	0.83	1.13	0.26	0.07	0.95	0.32	2.60	0.23
	-0.06	-0.72	3.13	0.29	1.63	1.21	1.67	1.04	3.71	-0.59	0.81	1.32	1.46	0.96	0.83	4.17	2.21	2.02
Lab 37	4.20	3.59	2.62	0.08	2.46	3.93	10.71	1.94	5.62	3.74	0.49	2.95	1.13	0.63	0.28	1.63	1.13	1.60
	3.72	1.20	1.56	-0.32	1.21	1.76	12.43	1.77	5.51	1.82	1.58	1.32	1.17	-0.32	0.26	1.99	2.13	1.56

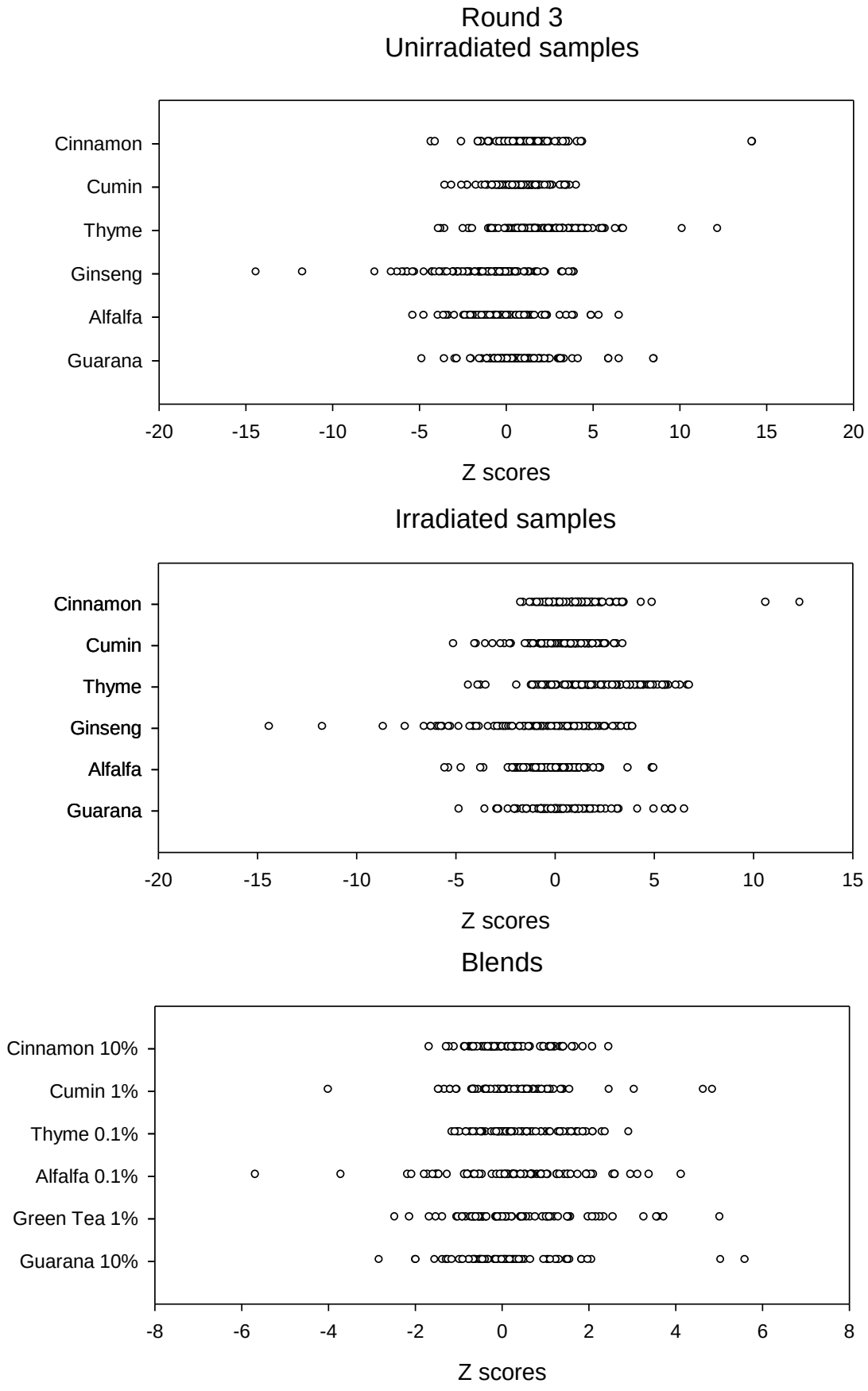


Figure 3.8 Participants' *z*-scores arranged by sample

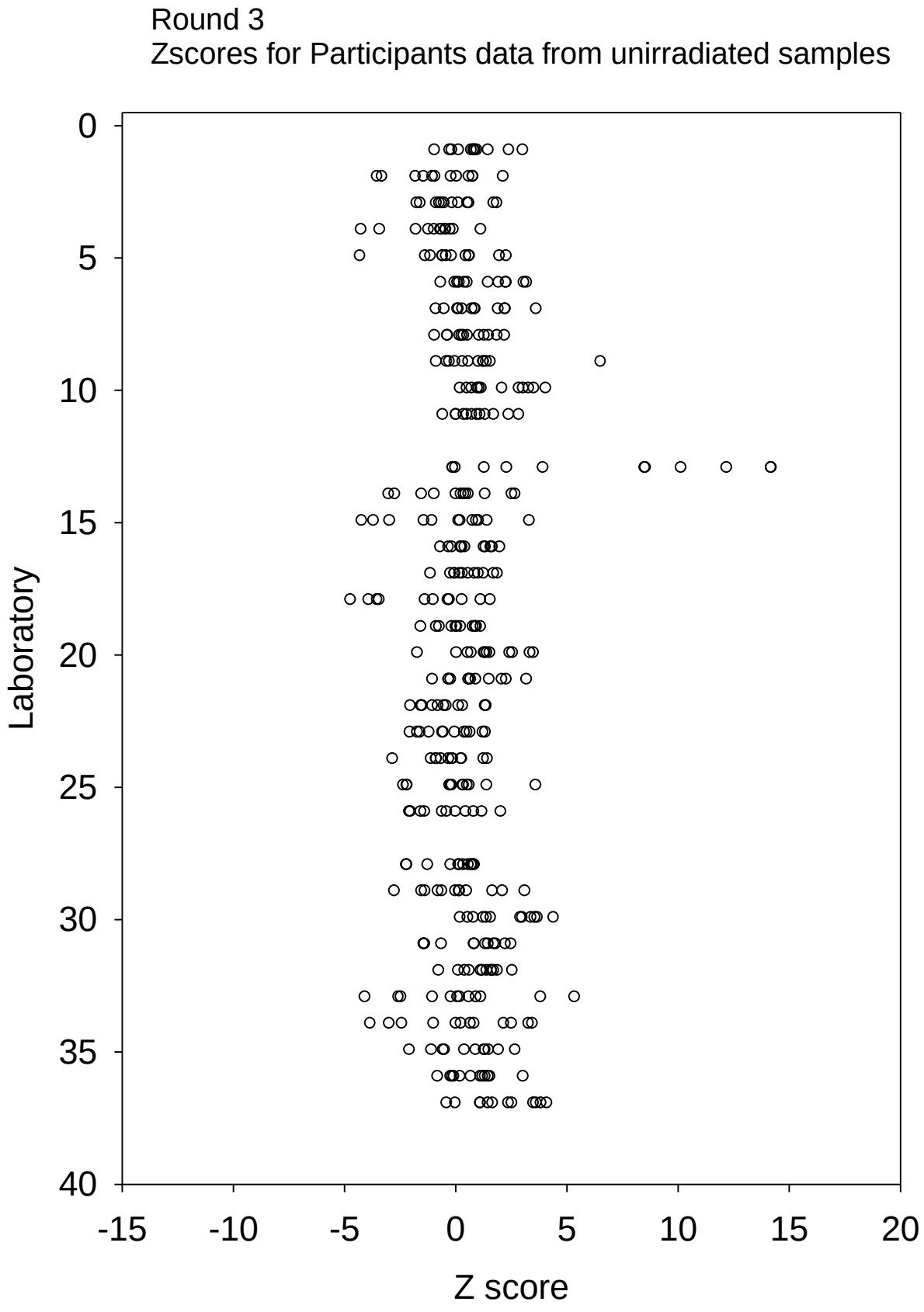


Figure 3.9 Participants' *z*-scores for unirradiated samples by laboratory

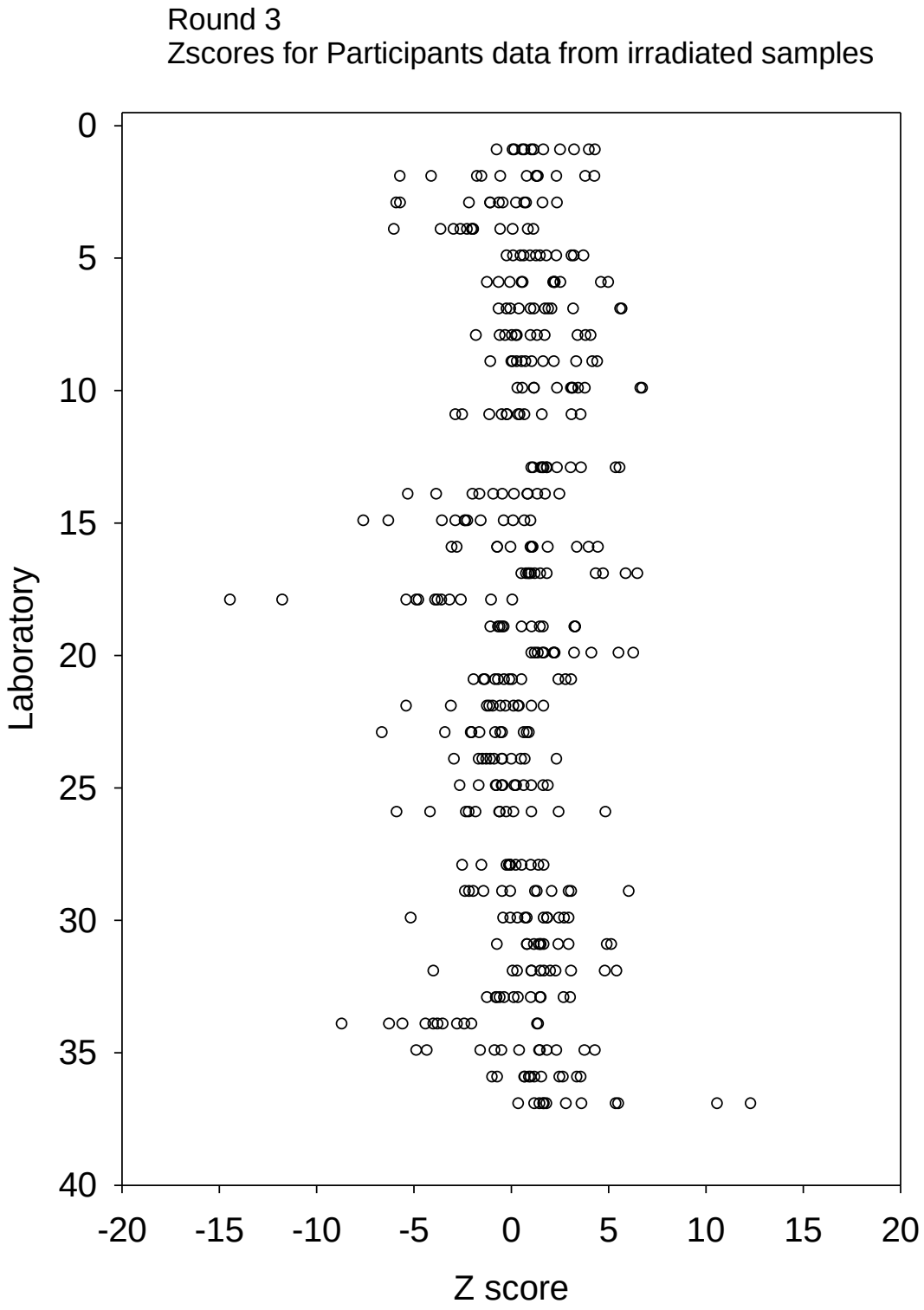


Figure 3.10 Participants' *z*-scores for irradiated samples by laboratory

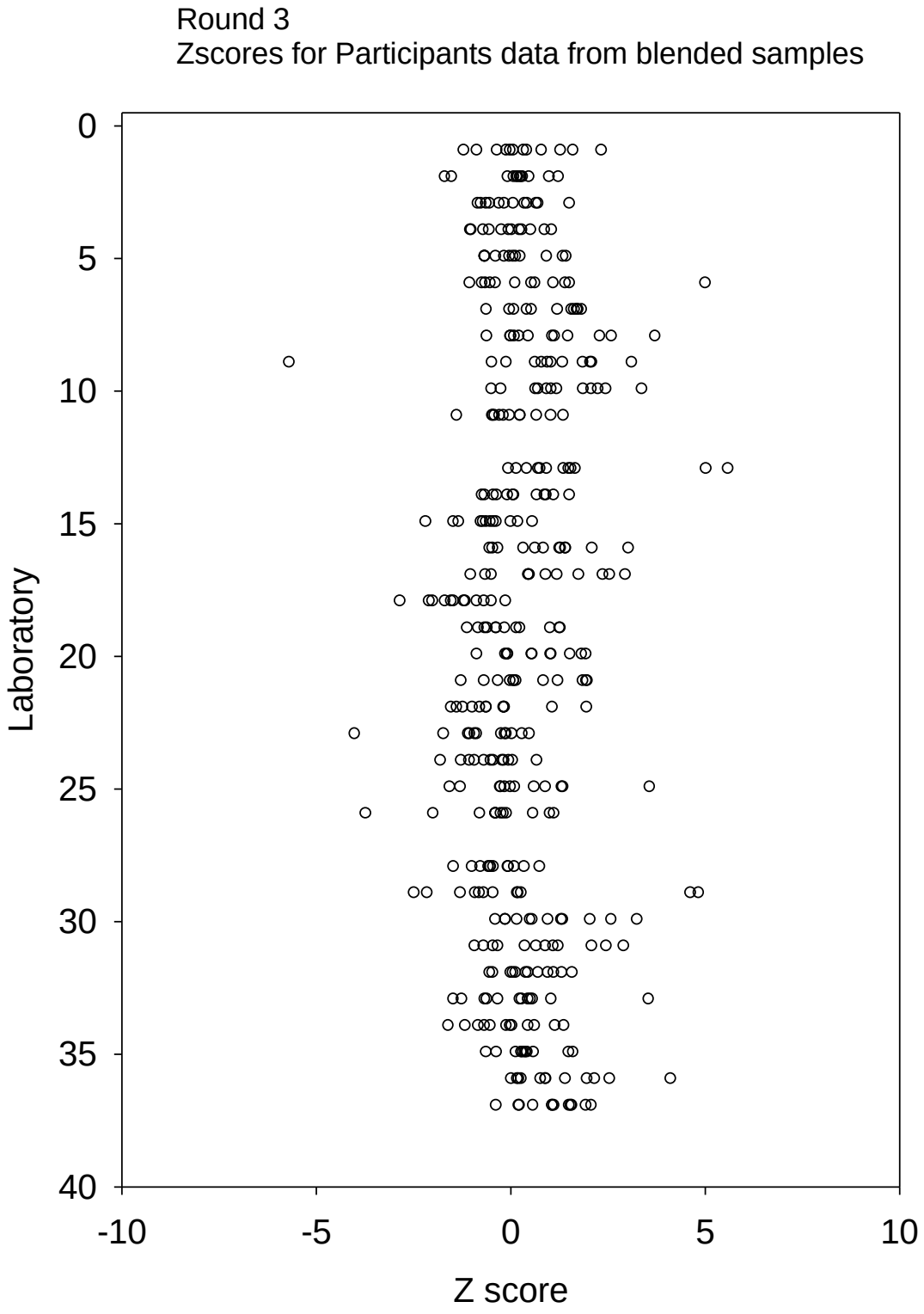


Figure 3.11 Participants' *z*-scores for blended samples by laboratory

Figures 3.12, 3.13 and 3.14 present participants *z*-scores in the form of 2-d colour contour plots. This forms of presentation permits visual identification of the interaction between test materials and participants with outlying results. With the reduced suite of only 18 samples this is a less suitable presentation method, but still yields some insights.

Figure 3.12 shows that lab 13 has contaminated at least 3 samples with irradiated material, which was observed in other figures above. The ginseng does appear to be "depressed" relative to other samples, as seen in the spread of its *z*-scores in Figure 3.8 for both unirradiated and irradiated materials, but the major contamination problems seen in earlier rounds appear to have been overcome.

Figure 3.13 shows that with the irradiated samples, almost all laboratories have elevated *z*-scores for thyme. This may be a consequence of the generally higher sensitivities of participants' instruments.

The contour plot for blends, figure 3.14, has fewer highs and lows, as is to be expected from the narrower range of *z*-scores.

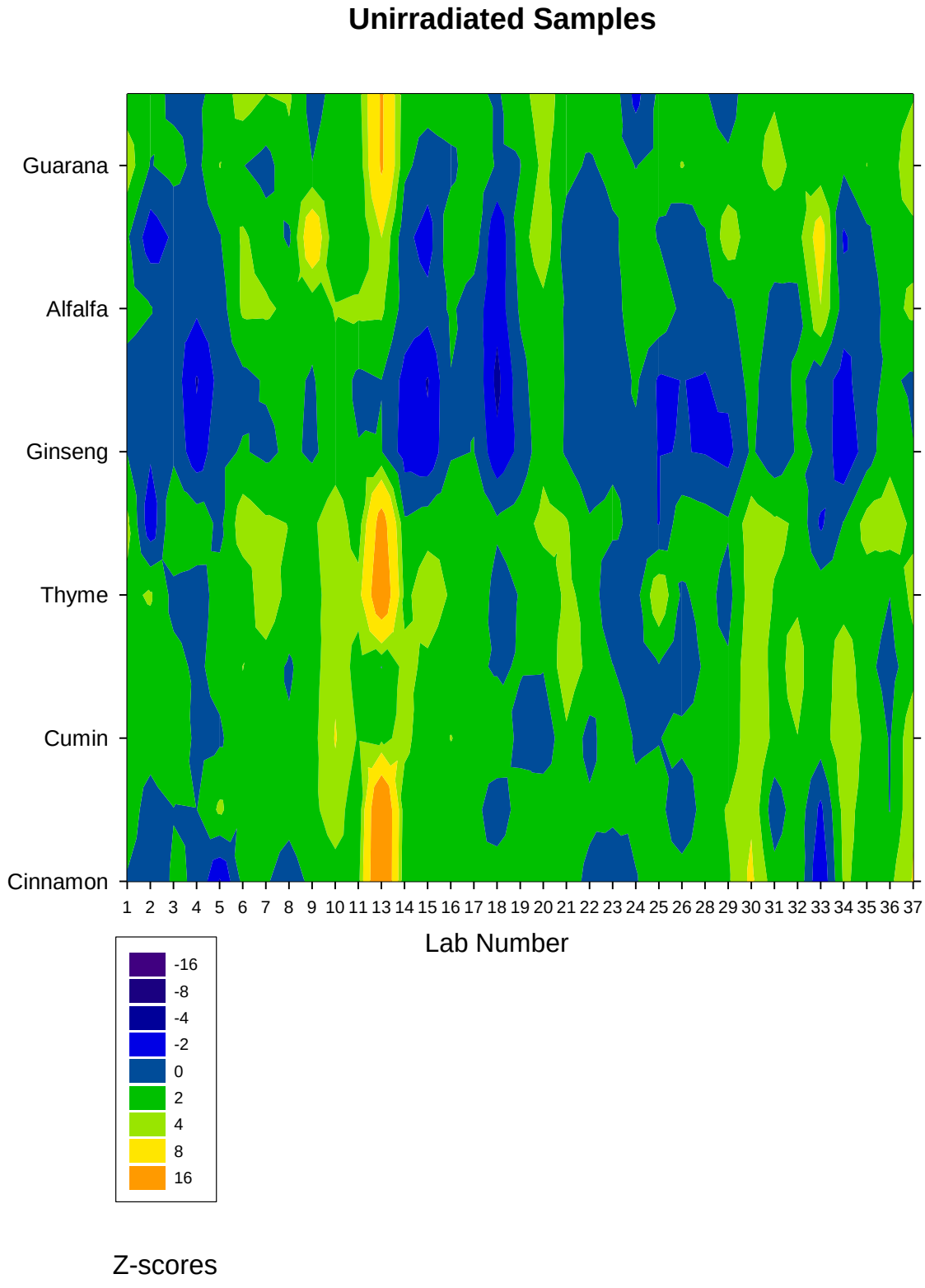


Figure 3.12 Contour plot for participants' data for unirradiated samples

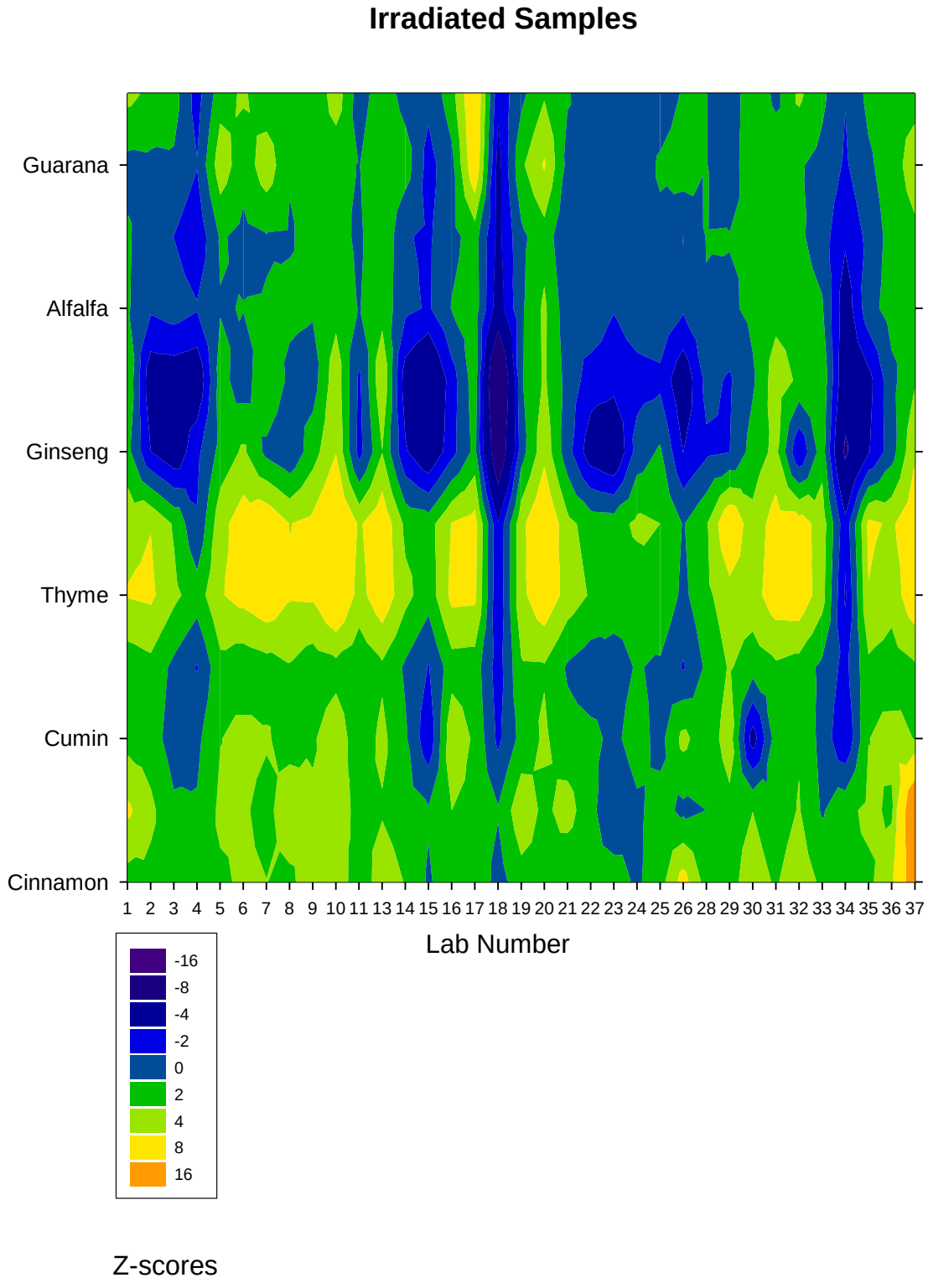


Figure 3.13 Contour plot for participants' data for irradiated samples

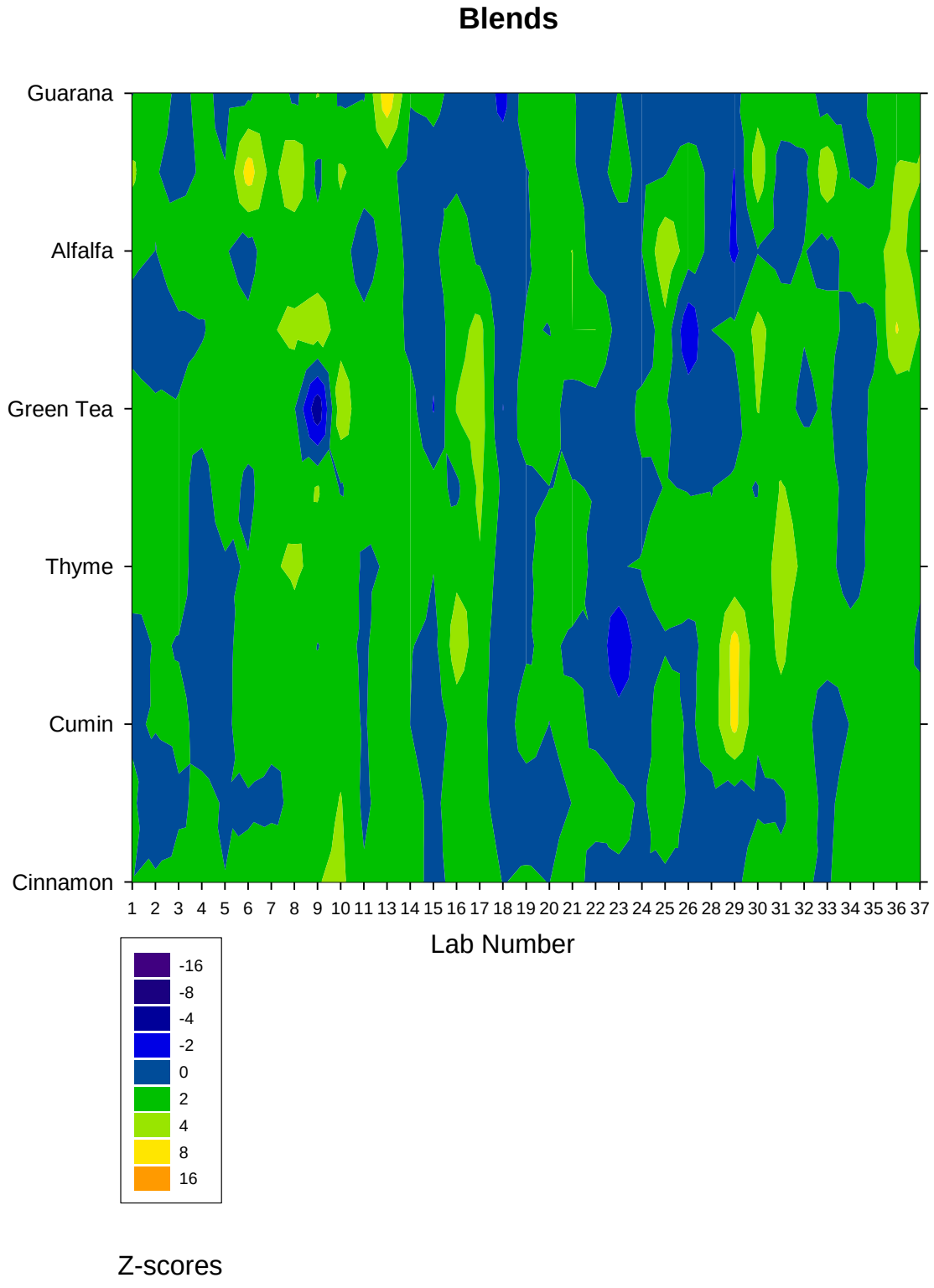


Figure 3.14 Contour plot for participants' data for blended samples

3.3.3.1 Pooled *z*-scores

In addition to separate *z*-scores for each sample and laboratory, pooled *z*—scores were also calculated. This approach had been briefly mentioned in Round 1.

	All products			Unirradiated			Irradiated			Blends		
	Mean z	SD z	Σz/√n	Mean z	SD z	Σz/√n	Mean z	SD z	Σz/√n	Mean z	SD z	Σz/√n
Lab 1	1.00	1.35	6.00	0.91	1.11	3.15	1.68	1.62	5.82	0.41	1.02	1.42
Lab2	-0.07	2.03	-0.41	-0.57	1.70	-1.96	0.25	3.00	0.87	0.11	0.86	0.38
Lab 3	-0.25	1.69	-1.50	-0.03	1.15	-0.09	-0.82	2.59	-2.83	0.09	0.71	0.33
Lab 4	-0.90	1.61	-5.39	-1.00	1.47	-3.46	-1.70	2.00	-5.88	0.00	0.69	0.01
Lab 5	0.59	1.51	3.52	-0.13	1.71	-0.44	1.70	1.29	5.90	0.18	0.74	0.64
Lab 6	1.25	1.67	7.50	1.31	1.31	4.55	1.80	1.93	6.25	0.63	1.64	2.20
Lab 7	1.35	1.53	8.07	1.05	1.31	3.65	1.95	2.09	6.75	1.03	0.86	3.58
Lab 8	1.02	1.39	6.09	0.72	0.96	2.51	1.23	1.86	4.27	1.09	1.28	3.78
Lab 9	1.13	1.95	6.77	1.12	1.90	3.87	1.58	1.75	5.48	0.69	2.22	2.38
Lab 10	2.17	1.69	13.03	2.05	1.34	7.10	3.10	2.09	10.73	1.37	1.13	4.74
Lab 11	0.48	1.36	2.88	1.04	1.00	3.60	0.32	1.94	1.11	0.08	0.75	0.28
Lab 13	3.57	4.00	21.41	6.34	5.67	21.96	2.68	1.55	9.28	1.69	1.81	5.84
Lab 14	-0.02	1.73	-0.09	0.09	1.81	0.30	-0.43	2.32	-1.50	0.30	0.75	1.04
Lab 15	-1.04	2.15	-6.24	-0.37	2.27	-1.28	-2.16	2.62	-7.49	-0.59	0.75	-2.03
Lab 16	0.88	1.57	5.28	0.75	0.88	2.58	0.93	2.42	3.21	0.97	1.07	3.36
Lab 17	1.41	1.75	8.44	0.62	0.87	2.14	2.64	2.20	9.13	0.97	1.30	3.35
Lab 18	-2.44	3.14	-14.67	-1.23	2.08	-4.27	-4.81	4.16	-16.65	-1.29	0.76	-4.48
Lab 19	0.45	1.23	2.68	0.23	0.85	0.79	1.07	1.65	3.72	0.04	0.82	0.13
Lab 20	1.67	1.64	10.02	1.51	1.46	5.22	2.85	1.72	9.87	0.65	0.88	2.27
Lab 21	0.59	1.35	3.55	0.92	1.25	3.20	0.31	1.71	1.08	0.54	1.07	1.87
Lab 22	-0.38	1.40	-2.29	-0.19	1.20	-0.64	-0.63	1.91	-2.17	-0.33	1.02	-1.16
Lab 23	-0.75	1.58	-4.50	-0.21	1.16	-0.72	-1.32	2.11	-4.56	-0.73	1.21	-2.52
Lab 24	-0.38	1.06	-2.26	-0.22	1.12	-0.76	-0.43	1.35	-1.50	-0.48	0.67	-1.66
Lab 25	0.16	1.43	0.98	0.05	1.68	0.16	0.03	1.31	0.10	0.41	1.36	1.43
Lab 26	-0.48	1.93	-2.87	-0.38	1.39	-1.30	-0.65	2.85	-2.27	-0.41	1.32	-1.41
Lab 28	-0.06	0.99	-0.36	-0.04	1.13	-0.12	0.15	1.16	0.53	-0.30	0.60	-1.03
Lab 29	0.38	2.18	2.26	0.15	1.65	0.50	0.82	2.58	2.84	0.16	2.32	0.57
Lab 30	1.44	1.71	8.63	2.32	1.42	8.03	0.94	2.18	3.25	1.06	1.15	3.66
Lab 31	1.22	1.57	7.35	0.75	1.48	2.59	2.10	1.69	7.27	0.83	1.26	2.87
Lab 32	1.16	1.57	6.97	1.22	0.90	4.21	1.74	2.40	6.02	0.53	0.68	1.84
Lab 33	0.38	1.84	2.30	0.24	2.63	0.82	0.67	1.41	2.31	0.25	1.31	0.85
Lab 34	-1.00	2.72	-5.99	0.33	2.43	1.16	-3.27	2.88	-11.34	-0.06	0.91	-0.19
Lab 35	0.52	1.83	3.11	0.68	1.38	2.36	0.41	2.88	1.42	0.46	0.64	1.61
Lab 36	1.24	1.24	7.41	0.87	1.05	3.02	1.50	1.44	5.18	1.34	1.22	4.64
Lab 37	2.47	2.67	14.85	2.17	1.50	7.51	4.16	3.83	14.40	1.10	0.75	3.81

Table 3.11 Pooled *z*-scores for participants for all products

3.2.4 Qualitative Results

As with earlier rounds, participants' results were classified into the 3 conventional screening bands (negative, intermediate and positive) using the two arbitrary threshold values (set at 700 and 5000 counts in keeping with BS EN 13751:2002) established during development of the method and in the validation studies. After examining the effect of standardisation to relative sensitivity for data from Round 2, no correction for this parameter was applied to Round 3 data, which are presented in descriptive form based on the data as received. The quantitative data as received were compared with the standard thresholds; instances where the participants used Calibrated PSL to inform their assessment are not included in this section. As in Round 2, duplicate aliquots are treated as separate outcomes.

Table 3.12 shows qualitative data compared with the thresholds and tabulated by participant. Table 3.13 shows them tabulated by test material. Table 3.14 shows the qualitative outcomes from round 3 in comparison with those obtained from rounds 1 and 2 and with all sets of reference data.

Overall the performance is again quite encouraging. No laboratory is clearly having problems, which is a marked improvement (Table 3.12). The effect of using different test materials and of introducing new laboratories is hard to assess, however.

When the ginseng is disregarded (Table 3.13), only 2 aliquots of unirradiated material produced positive terminal counts (0.5%), which is a significant improvement. It is still the case that a significant proportion (nearly 25%) of unirradiated samples exceeded the lower threshold. This underlines the importance of further investigation of intermediate results.

For irradiated samples, only a single aliquot failed to reach the lower threshold, but 17% were between the two thresholds. This implies low mineral yield or sensitivity; the non-positive results almost exclusively coming from cinnamon (Table 3.13). Again further investigation is indicated.

For the blends, the results seem to be strongly product-related, with one of the 10% blends (cinnamon, of low sensitivity) not being picked up except in 2 aliquots whereas the 10% guarana exceeded the lower threshold in almost all cases. In contrast, thyme with low concentration (0.1%) but high sensitivity and the 1% green tea yield very few negative results. This implies, for this particular set of 6 materials at least, that sensitivity dominates concentration in determining PSL outcomes.

Table 3.14 compares qualitative outcomes from all 3 rounds and with the different reference sets (Round 1 all irradiated and unirradiated materials used in the first 2 rounds, Round 2 the addition of blending and Round 3 a complete new set of analyses). The proportion of unirradiated samples which should be investigated further remains at about 30%. Successful detection of irradiated materials is still essentially 100%. Blends are undoubtedly problematic. The 2 rounds of the study which included them and the 2 associated sets of reference data all reveal that 30-40% of blends will not be referred for further analysis. All analytical methods have limits and blends test those limits for PSL.

	Unirradiated			Irradiated			Blends		
Lab	N	I	P	N	I	P	N	I	P
1	7	3	2	0	2	10	5	5	2
2	8	2	2	0	2	10	4	8	0
3	8	2	2	0	2	10	3	9	0
4	8	2	2	0	3	9	7	5	0
5	7	3	2	0	2	10	6	5	1
6	7	3	2	0	2	10	3	9	0
7	6	4	2	0	2	10	2	7	3
8	7	3	2	0	2	10	3	7	2
9	8	2	2	0	2	10	4	5	3
10	6	4	2	0	2	10	0	10	2
11	8	2	2	0	2	10	5	6	1
13	2	6	4	0	2	10	3	7	2
14	7	3	2	0	2	10	4	7	1
15	8	2	2	0	3	9	6	6	0
16	8	2	2	0	2	10	3	7	2
17	8	2	2	0	2	10	4	4	4
18	8	2	2	0	4	8	9	3	0
19	8	2	2	0	2	10	5	7	0
20	6	4	2	0	2	10	2	9	1
21	7	3	2	0	2	10	4	6	2
22	8	2	2	0	2	10	6	5	1
23	8	2	2	1	1	10	6	6	0
24	8	2	2	0	2	10	6	6	0
25	7	3	2	0	2	10	5	7	0
26	7	3	2	0	2	10	5	7	0
28	8	2	2	0	2	10	4	8	0
29	8	2	2	0	2	10	4	6	2
30	4	6	2	0	2	10	3	7	2
31	6	4	2	0	2	10	4	6	2
32	8	2	2	0	2	10	4	6	2
33	8	2	2	0	2	10	4	8	0
34	6	4	2	0	2	10	3	9	0
35	7	3	2	0	2	10	3	9	0
36	8	2	2	0	2	10	2	9	1
37	3	7	2	0	0	12	3	7	2
Total	246	102	72	1	71	348	144	238	38
Percentage	58.57%	24.29%	17.14%	0.24%	16.90%	82.86%	34.29%	56.67%	9.05%

Table 3.12 Participants' qualitative results by laboratory

	Unirradiated		
	N	I	P
Cinnamon	65	3	2
Cumin	61	9	0
Thyme	66	4	0
Ginseng	0	0	70
Alfalfa	0	70	0
Guarana	54	16	0
Total	246	102	72
Percentage	58.57%	24.29%	17.14%
Total without ginseng			2
Percentage without ginseng	73.14%	29.14%	0.48%

	Irradiated		
	N	I	P
Cinnamon	1	67	2
Cumin	0	0	70
Thyme	0	0	70
Ginseng	0	0	70
Alfalfa	0	0	70
Guarana	0	4	66
Total	1	71	348
Percentage	0.24%	16.90%	82.86%

	Blends		
	N	I	P
Cinnamon	68	2	0
Cumin	24	44	2
Thyme	5	45	20
Green tea	1	55	14
Alfalfa	42	28	0
Guarana	4	64	2
Total	144	238	38
Percentage	34.29%	56.67%	9.05%

Table 3.13 Participants' qualitative results by product

		Round 1 and 2 reference		Round 1 participants'		Round 2 participants'		Round 3 reference		Round 3 participants'	
		Total	%	Total	%	Total	%	Total	%	Total	%
U	Neg	411	83.9	1107	72.9	1478	74.6	79	65.8	246	58.6
	Int/Pos	79	16.1	412	27.1	502	25.4	41	34.2	174	41.4
I	Neg	0	0	5	0.3	2	0.1	2	1.7	1	0.2
	Int/Pos	490	100	1514	99.7	1978	99.9	118	98.3	419	99.8
B	Neg	23	38.3			103	28.6	50	41.7	144	34.3
	Int/Pos	37	61.7			257	71.4	70	58.3	276	65.7

Table 3.14 Comparison of participants' qualitative percentage with the reference set

Figure 3.15 shows negative and intermediate qualitative outcomes plotted against pooled *z*-score for unirradiated materials. The two plots are almost perfect mirror images of one another since there are almost no positive results for unirradiated materials apart from the ginseng. As in Round 1, there is a general tendency for higher pooled *z* to be associated with higher percentages of non-negative outcomes but with so few samples this is not a convincing trend.

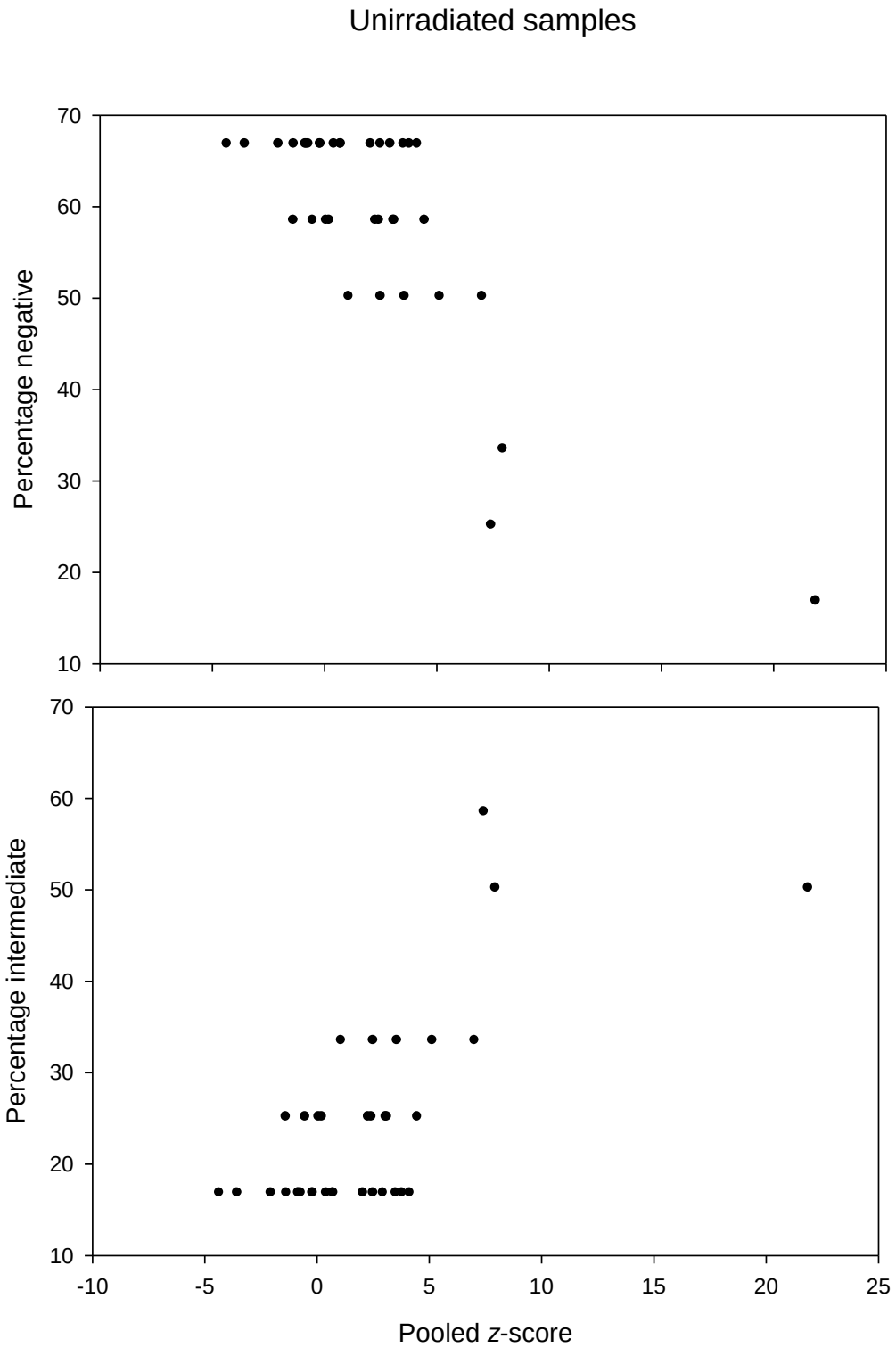


Figure 3.15 Pooled *z*-score vs qualitative percentage

4. CALIBRATED PSL

4.1 Calibrated PSL Homogeneity Testing

Round 2 introduced Calibrated PSL measurements for those laboratories able to irradiate their material, and Round 3 continued this. 10 of the 35 labs who performed screening also calibrated their samples. To provide reference data, Calibrated PSL was performed at SUERC in May and June 2007 using the PSL geometries screened on the DOS system. A dose of approximately 1kGy was applied to the samples a week prior to the second read-out. For both DOS and Windows systems screening samples had been prepared in thin-layer format to minimise mixing between the read-outs. This was not done in Round 2, where thick preparations were used.

4.2 Homogeneity Testing Results

The PSL terminal counts for the Calibrated measurements are fully tabulated in Appendix B. Figure 4. 1 presents the data as a scatter plot with initial PSL against Calibrated PSL; the data show good separation of irradiated and unirradiated products, with the blends overlapping both categories as expected. There are fewer samples and therefore fewer data points than in Round 2, which contributes to the clearer separation of the irradiated samples into clusters, although they still fall along an oblique line. The ginseng is clearly separated from the other products, in both irradiated and unirradiated form, representing the highest sensitivity cluster in the irradiated product grouping, and also the highest group of samples in the initial PSL reading in figure 4.1. This sample is considered to have contained significant proportionbs of irradiated material on arrival. The other materials are segregated on the basis of sensitivity, although this is slightly obscured by the location of the results from the blends. Again it is clear that Calibrated PSL does not reliably distinguish blends even when the irradiated and unirradiated portions are the same material.

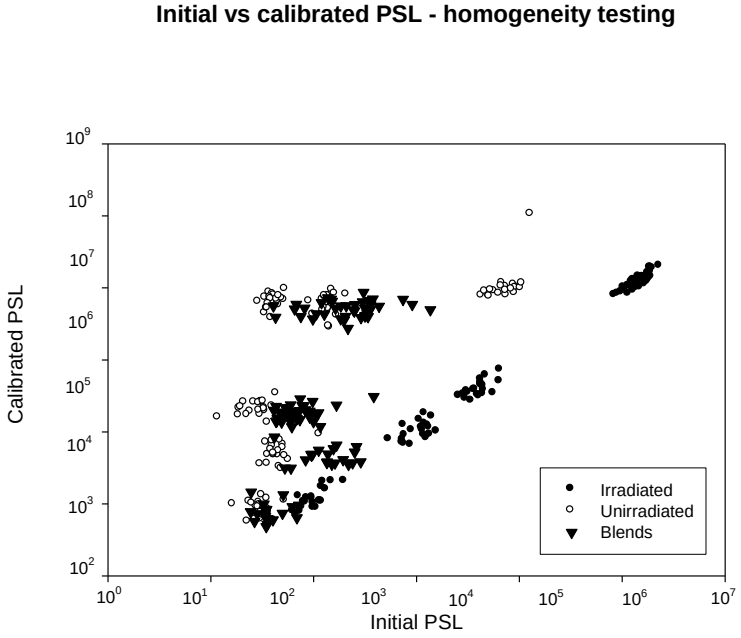


Figure 4.1 Initial vs Calibrated PSL for reference materials

4.3 Participants' Results

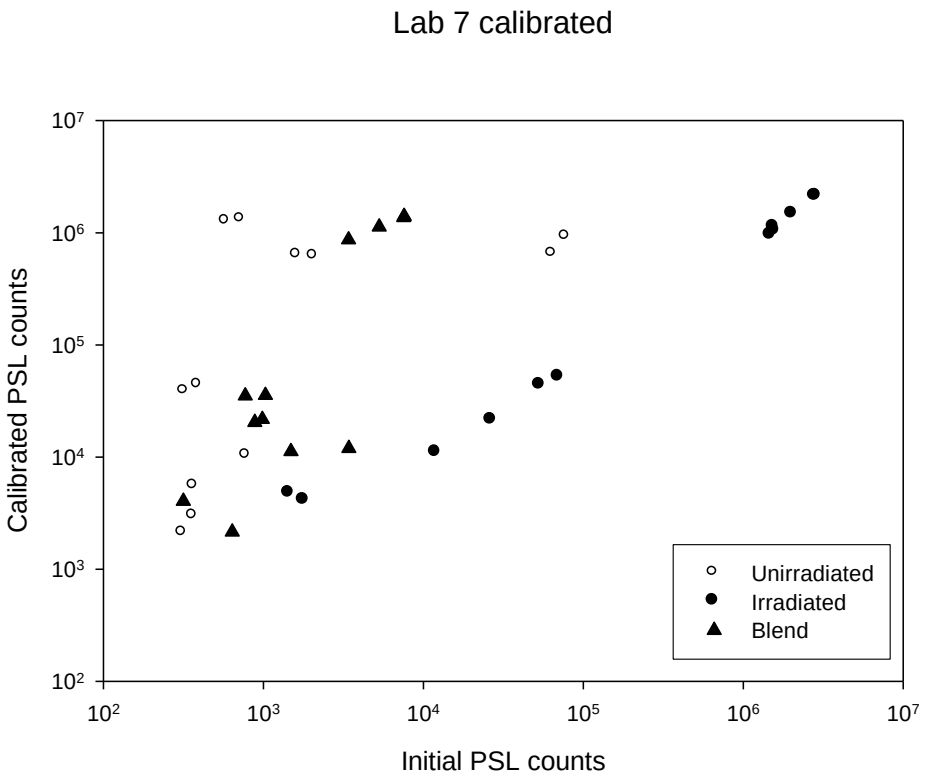
Participants returned raw data for their calibrated measurements. In some cases it was clear that they had used the sensitivity information provided by the calibration to reach a conclusion about the sample status. In other cases they may have done this but did not explicitly say so. It is not clear whether thick or thin preparations were used. In Round 2 requests for more petri-dishes implied that some laboratories had not followed the correct procedure of irradiating the geometries they had already screened, but had re-dispensed samples for calibration. It is not clear whether this also happened in Round 3. For each of the 10 laboratories which returned calibrated data, a plot equivalent to Figure 4.1 was produced. These are displayed below as Figures 4.2 – 4.6. Full data are in Appendix C.

Table 4.1 shows the type of source used by participants, where this was stated in their returns. It also shows whether they used the information obtained from the second measurement to inform their judgment about the samples.

Lab	Type of source	Cal results used in classification?
7	^{60}Co	Not stated
16	LINAC 10MeV	Not stated
21	^{60}Co (Gamma cell 220 Nordion)	Not stated
23	x-ray, 50 kV, 50W	Yes; not clear if conclusions changed
24	gamma from ^{60}Co	Not stated
25	^{60}Co	Not stated
28	^{60}Co	Not stated
31	^{60}Co	Not stated
34	^{60}Co	Yes; clear which conclusions changed
35	^{60}Co	Yes; clear which conclusions changed

Table 4.1 Type of source used for calPSL and influence on classification

In comparison with Round 2 participants' data, Round 3 results show sparse trends lines as a result of the reduced number of samples. However the underlying trends of the irradiated and unirradiated zones on the calibrated plots appear to be represented. As before the blends are not easily distinguishable from unirradiated samples. Again, in a similar manner to the reference data participants results from ginseng suggest the presence of irradiated material. In the round 2 report calibrated PSL were presented and visual evidence of differing levels of dispersion noted. Prior to round 3 further consideration has been given to additional methods for analysing and comparing data of this sort. An approach based on regression analysis of the unirradiated and irradiated sample types coupled to examination of prediction intervals around the trend lines has been worked out. For irradiated samples the slope of the trend line should indicate the combination of dose response of the materials and dose-difference between the initial irradiation and the calibrating dose, coupled to fading or bleaching between the first two readings. Dispersion around this trend line may also reflect sample handling; while the breadth of the similar trend data for unirradiated samples is also expected in incorporate variations due to natural residual geological PSL. At low sensitivities there will generally be an intersection between the lower prediction interval for unirradiated and irradiated samples and the upper prediction interval of the irradiated samples. Beneath these limits on the calibrated PSL plot a "zone of ambiguity", named ZOA can be defined. This is expected to show interlaboratory variation. Regression analysis and ZOA analysis have been explored with round 2 data and will be applied to these results



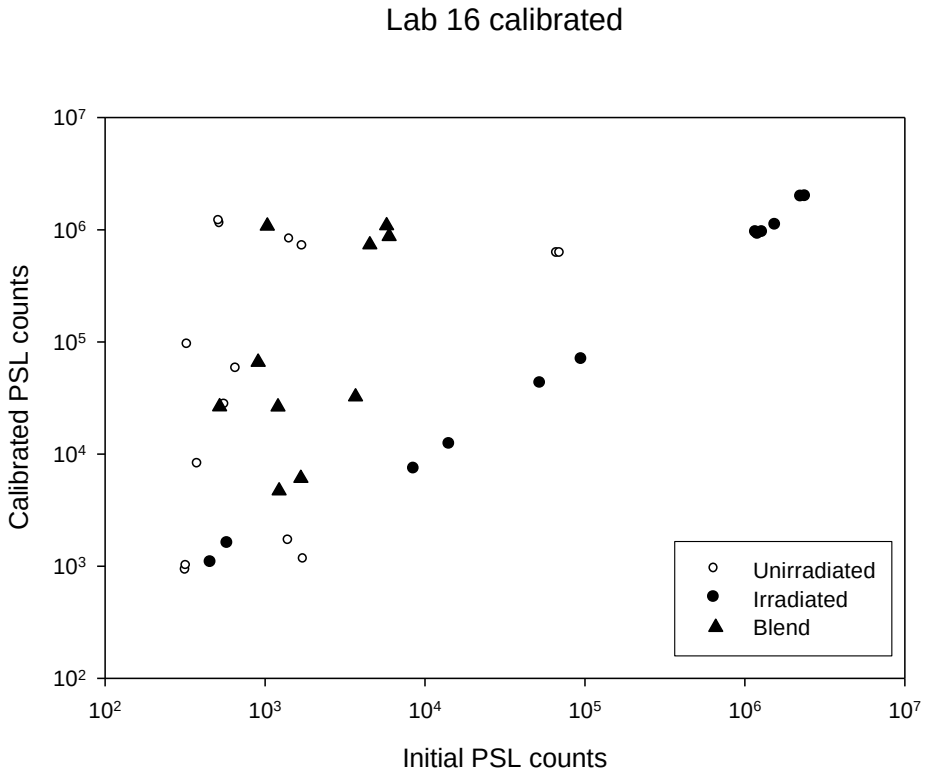
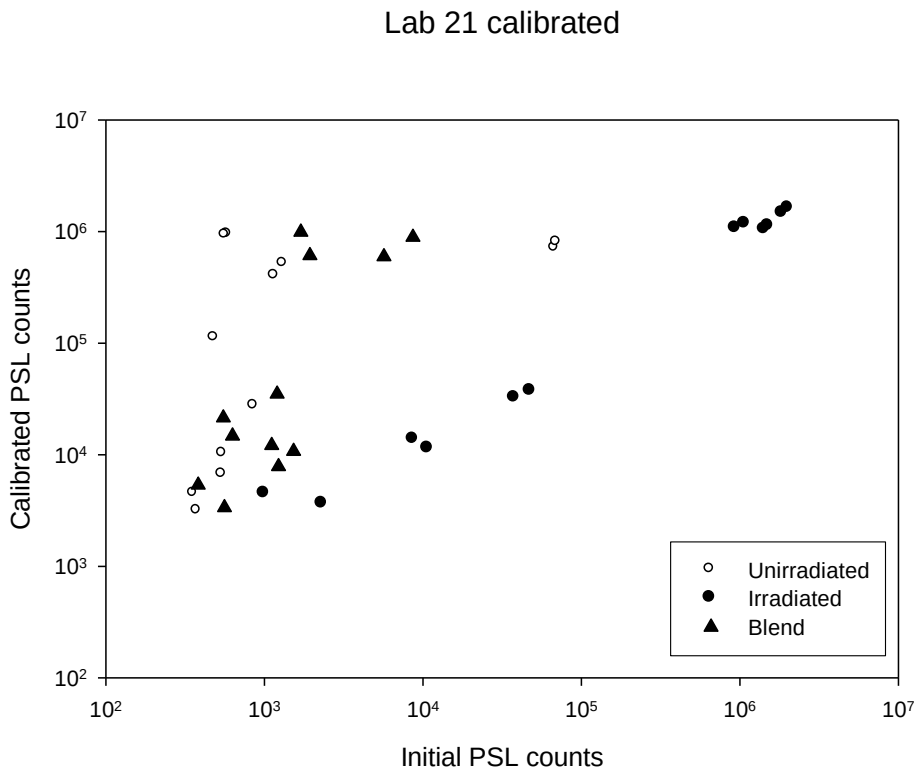


Figure 4.2 Initial vs Calibrated PSL for laboratories 7and 16



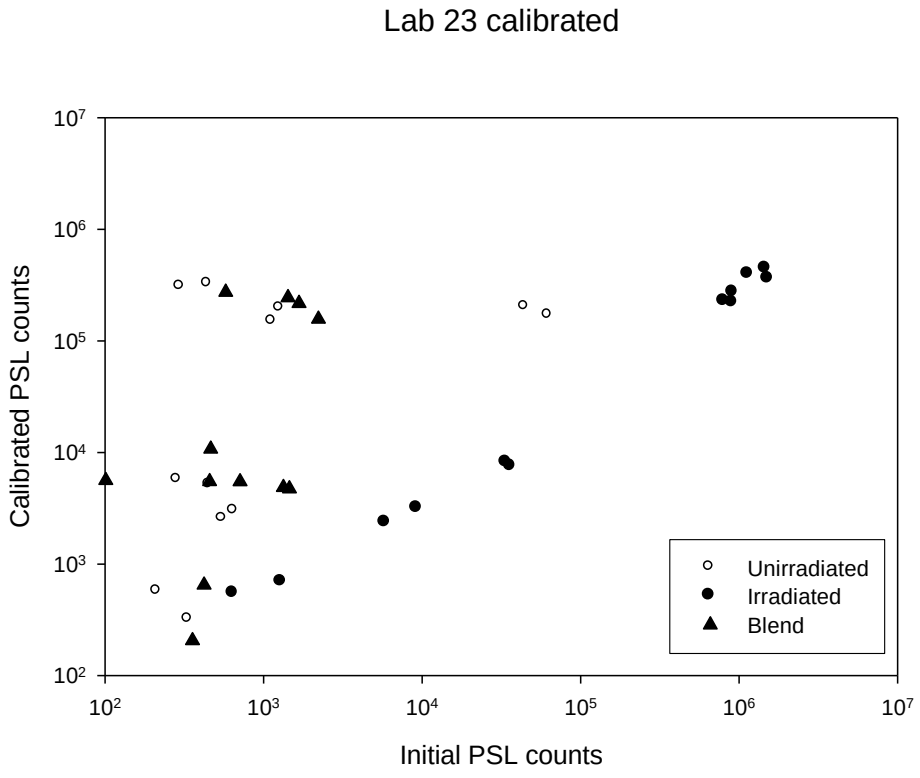
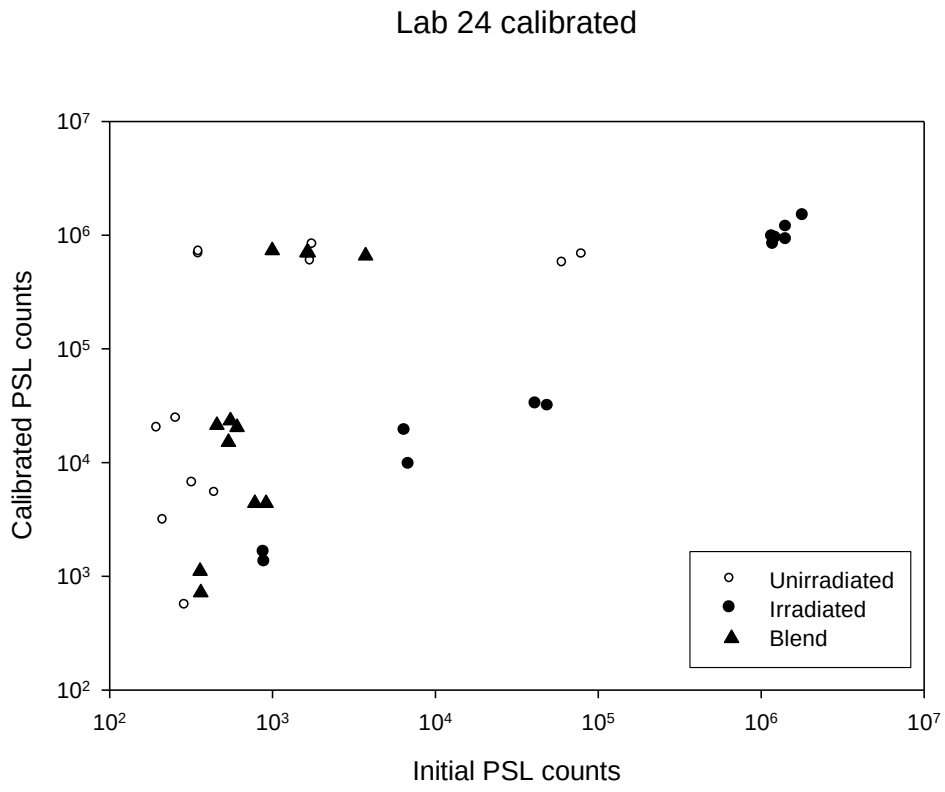


Figure 4.3 Initial vs Calibrated PSL for laboratories 21 and 23



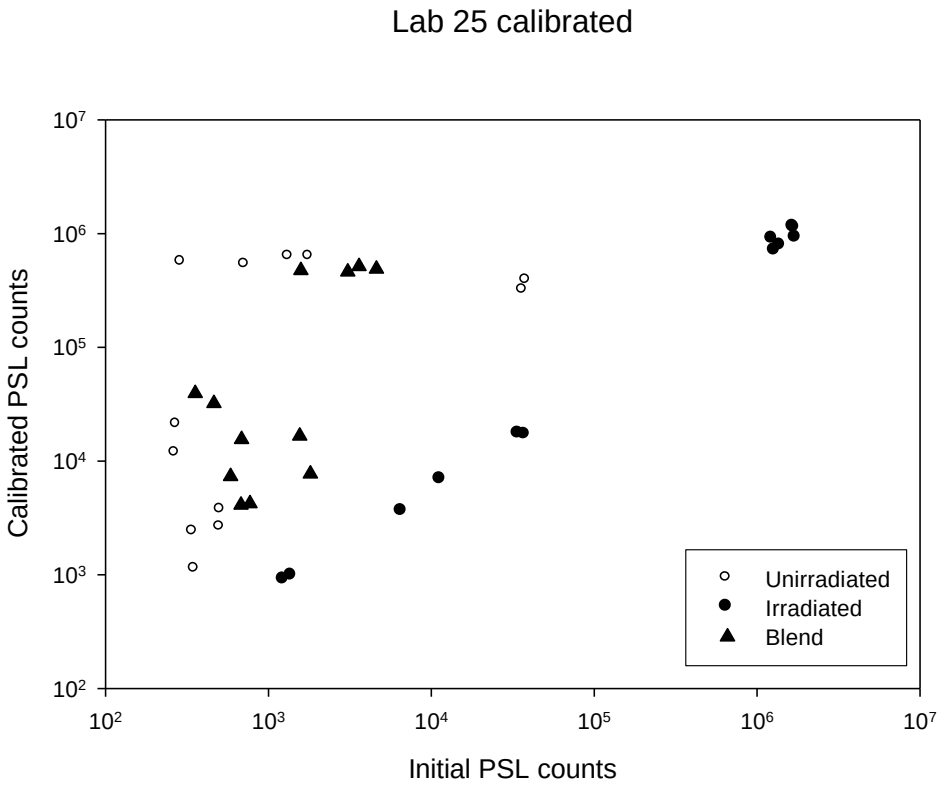
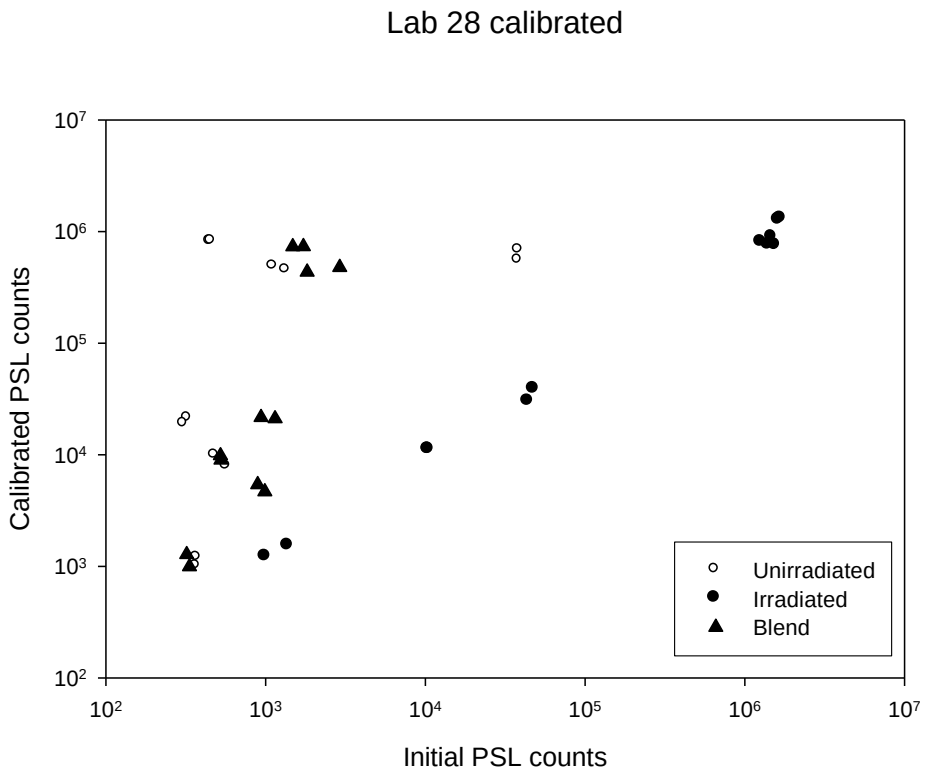


Figure 4.4 Initial vs Calibrated PSL for laboratories 24 and 25



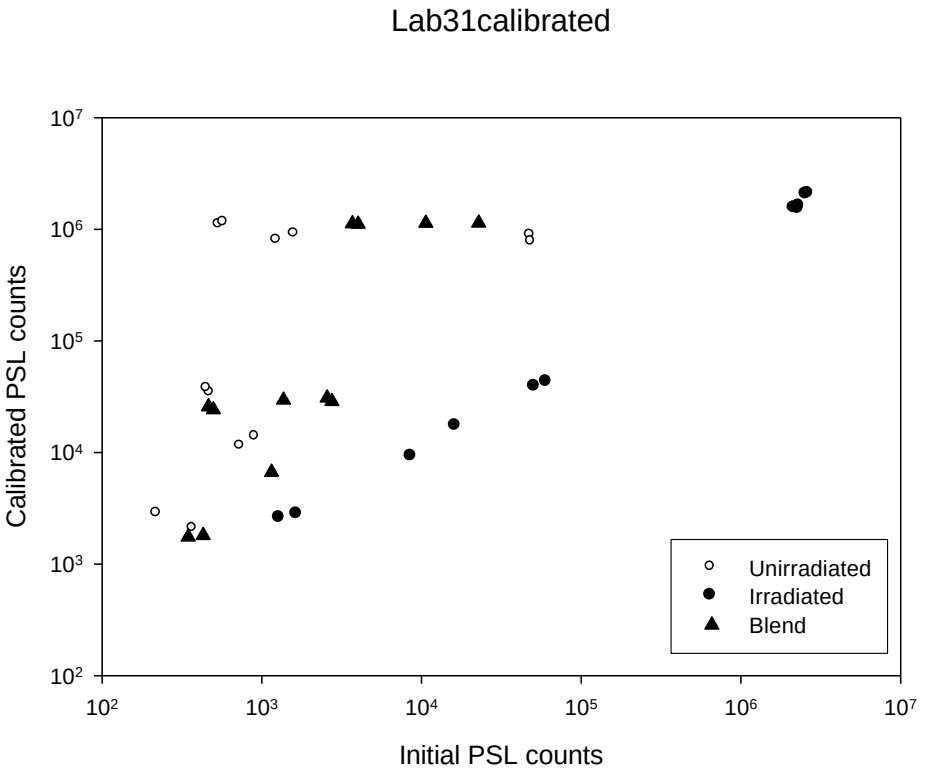


Figure 4.5 Initial vs Calibrated PSL for laboratories 28 and 31

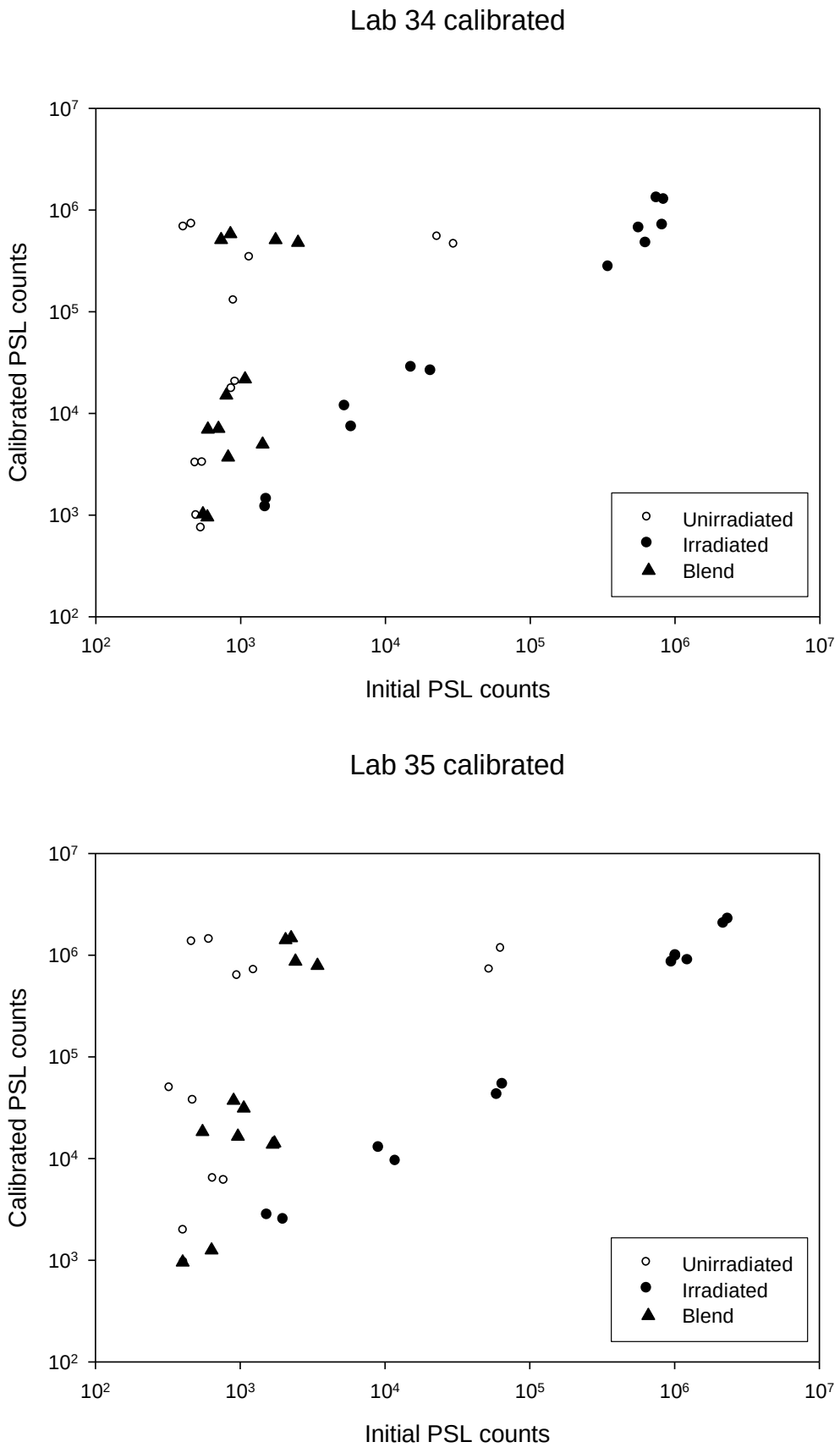


Figure4.6 Initial vs Calibrated PSL for laboratories 34 and 35

5. THERMOLUMINESCENCE ANALYSIS

5.1 TL Homogeneity Testing

As for PSL, new homogeneity testing was required for Round 3. This was conducted at SUERC in May and June 2007. For irradiated and unirradiated products sent to participants, 2 discs were prepared from each of 10 pots; 20 discs were prepared from each of the blended materials. To ensure that there was no cross-contamination between irradiated, unirradiated and blended samples, the unirradiated products were separated and read out first, then the blended products and finally the irradiated products; the procedure was carried out with the usual quality assurance steps using process and glassware blanks and temperature calibration checks (CaF_2).

5.2 TL Homogeneity Testing Results

EN1788 calls for TL quantification of glow 1 signals, glow 2 signals, the glow ratio and identification of whether or not a peak can be observed in the 150-250°C region. Where glow 2 intensities are less than 10 times the laboratory minimum detectable level, defined by the mean and 3 standard deviations of full-process blanks the mineral yield is considered unsatisfactory. An additional criterion for use in UK surveys, which has yet to be added to EN1788, was introduced in 2001 which requires any sample where the presence of irradiated material has been identified on the basis of a low temperature peak in glow 1 to have intensity which exceeds the minimum detectable level.

Figures 5.1-5.3 are scatter plots of glow 1 against glow 2 log intensities (integrated for 220°-240°C) for each product, with the three irradiation categories indicated by different symbols. These plots show complete separation of irradiated and unirradiated materials. For the blends, the degree of separation varies. Thyme and Green Tea are well separated (in the latter case the material differs from the irradiated and unirradiated samples so separation is less significant). The Guarana blend is not distinguishable from the irradiated material; this blend is at 10% and a high sensitivity material. For Cinnamon, Cumin and Alfalfa the blends are interspersed with the unirradiated samples. These blends cover all three concentrations, suggesting as in the PSL results that sensitivity is dominant.

Figures 5.4-5.6 plot glow ratio for the same temperature interval, again showing very good discrimination between irradiated and unirradiated samples, with mixed results from the blends. Guarana and Thyme blends are well separated, Ginseng/Green Tea remains a special case (but with good separation) and the other 3 products do not show segregation for the blends, which mingle with the unirradiated samples. Table B28 in Appendix B summarises the reference values of glow ratio, spreadsheets with G1 and G2 intensities, G1/G2 ratios and peak classifications have been prepared. As can be seen in figures 5.3 and 5.5 the untreated ginseng gives glow ratios generally above the EN1788 threshold of 0.1 for indetifying irradiated material; in the majority of analyses there is evidence of a low temperature peak in G1. These observations, together with the PSL data referred to above, confirm the sample as a mixture containing irradiated material.

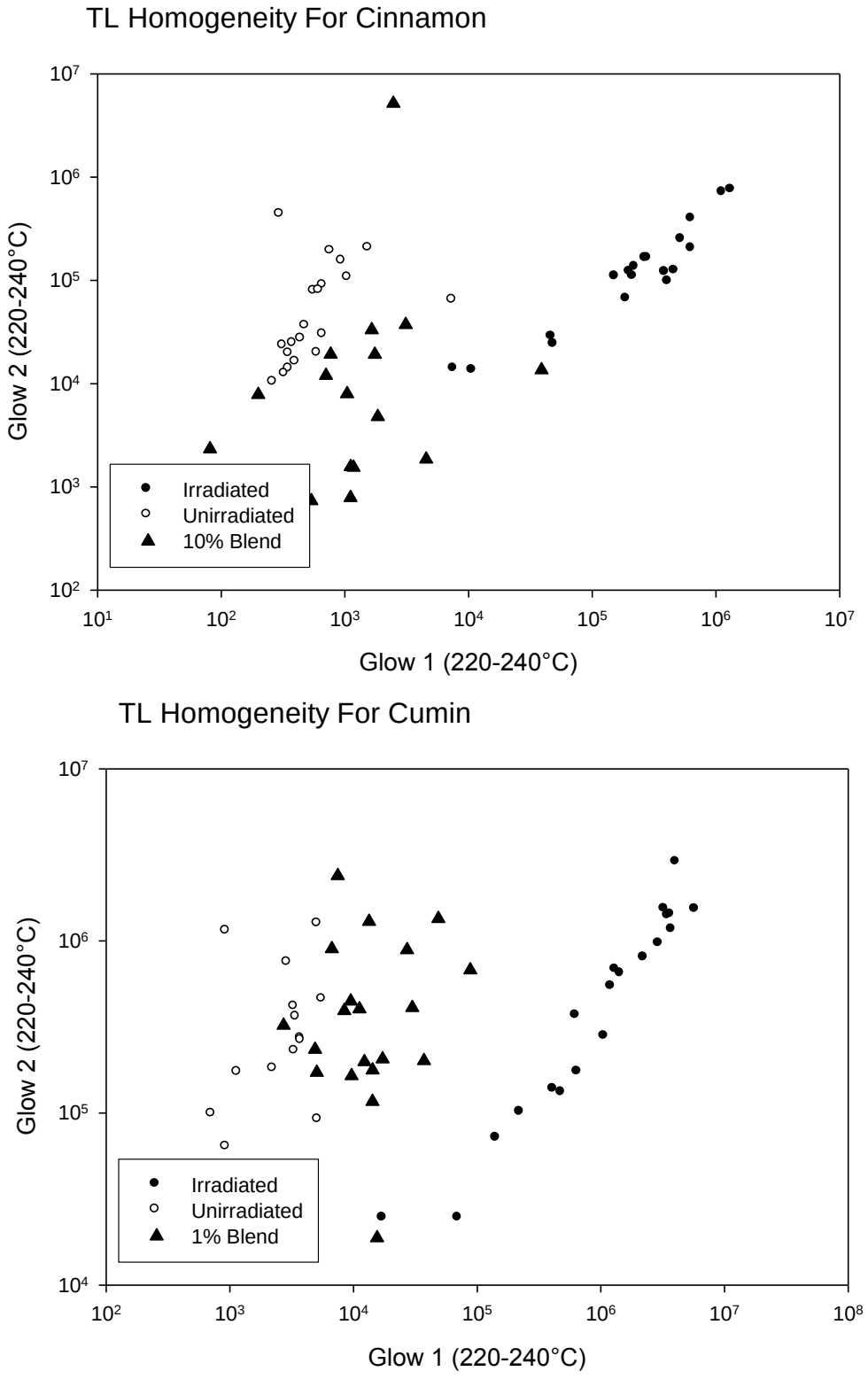


Figure 5.1 TL homogeneity testing glow 1 vs glow 2 scatter plot for cinnamon and cumin

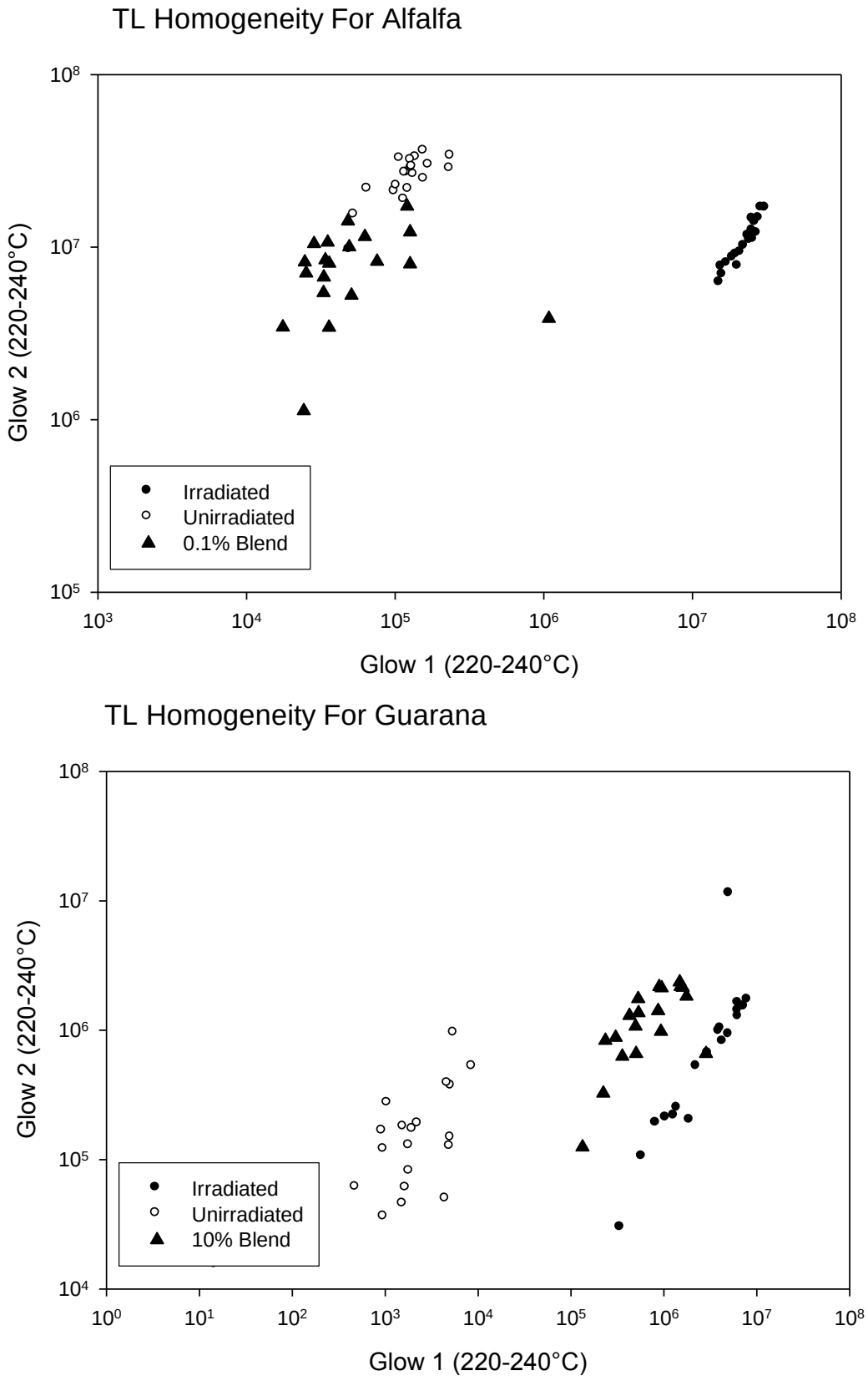


Figure 5.2 TL homogeneity testing glow 1 vs glow 2 scatter plot for alfalfa and guarana

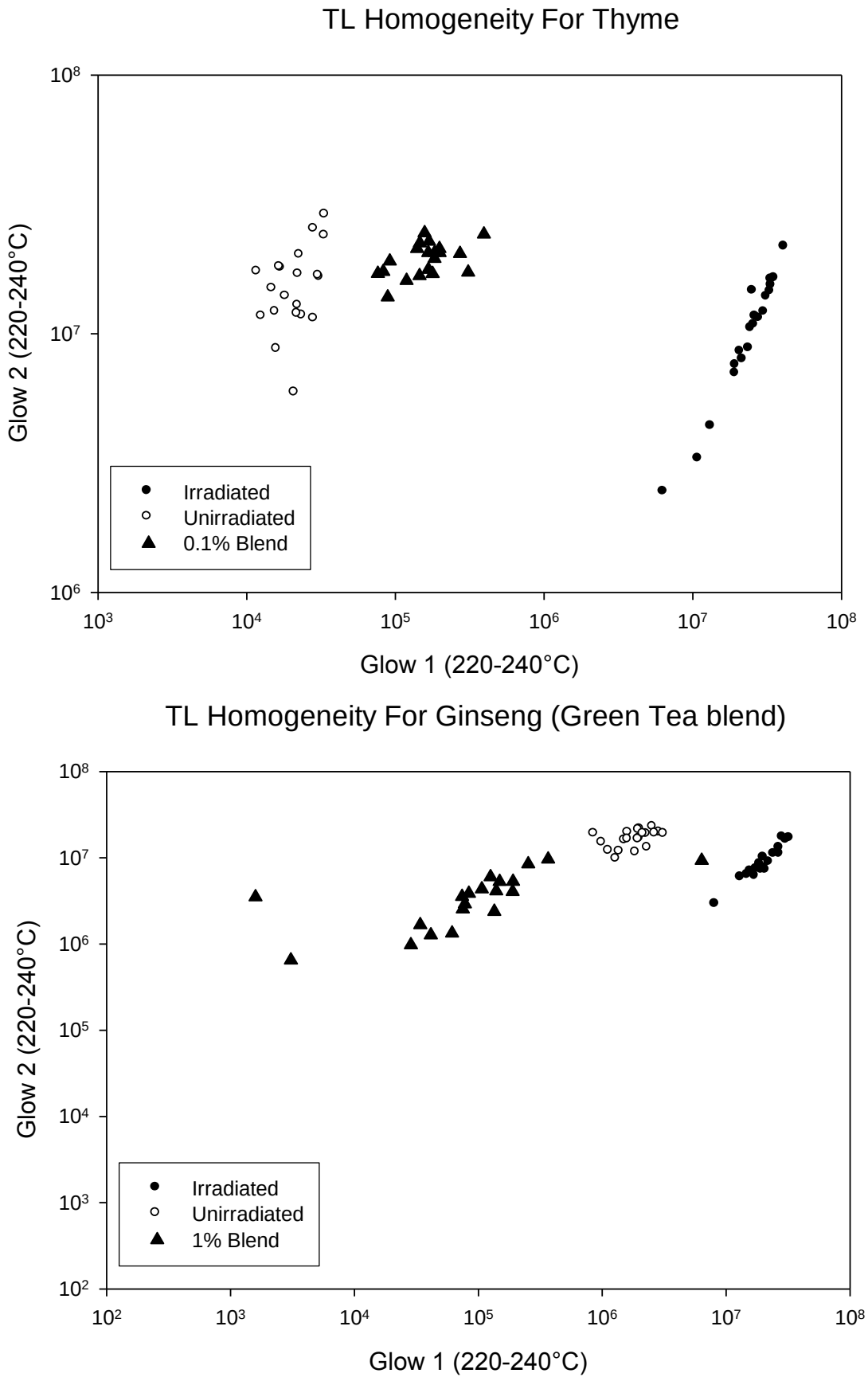
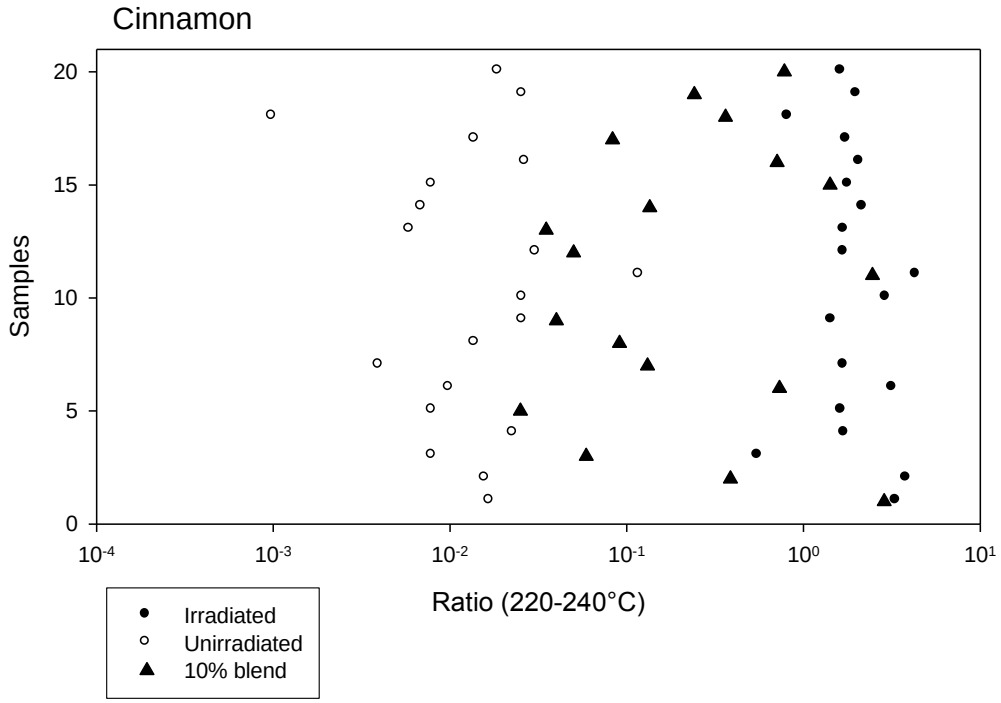


Figure 5.3 TL homogeneity testing glow 1 vs glow 2 scatter plot for thyme and ginseng (green tea)



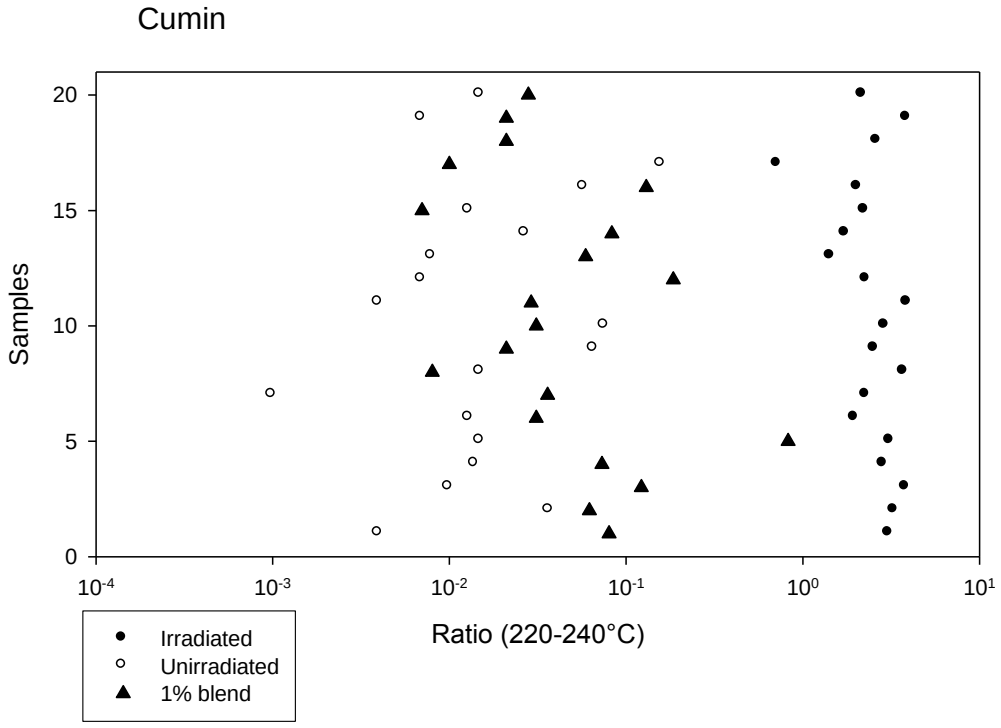
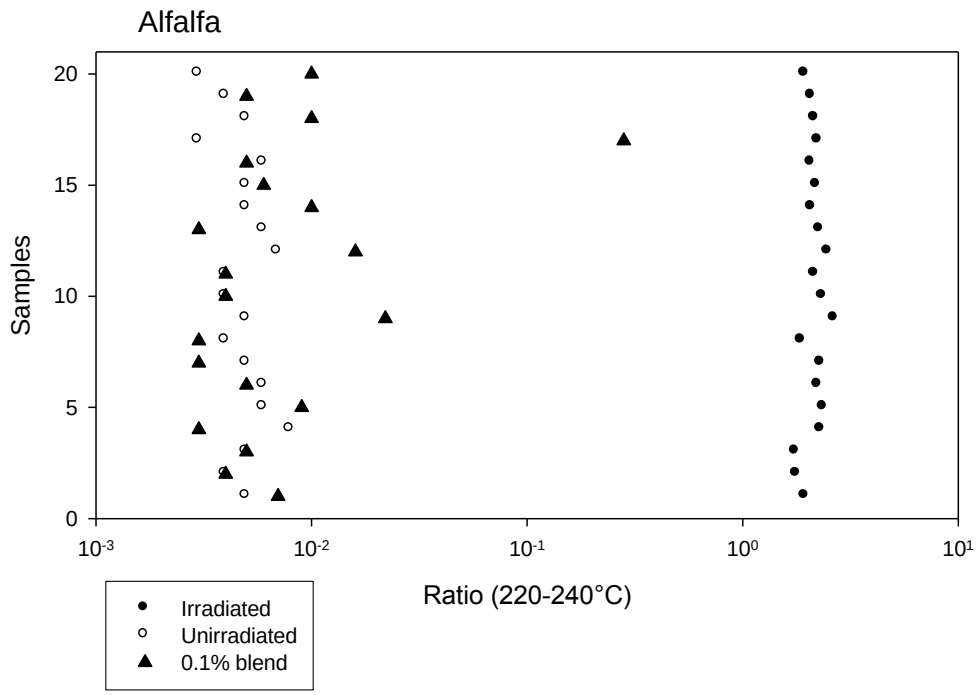


Figure 5.4 TL homogeneity testing glow ratio plot for cinnamon and cumin



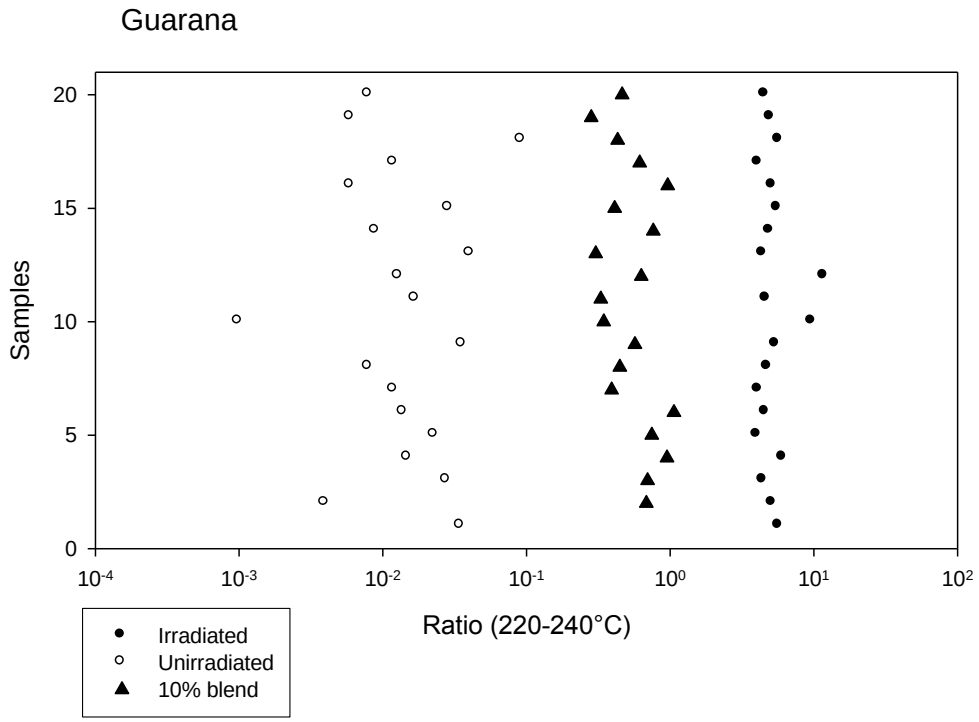


Figure 5.5 TL homogeneity testing glow ratio plot for alfalfa and guarana

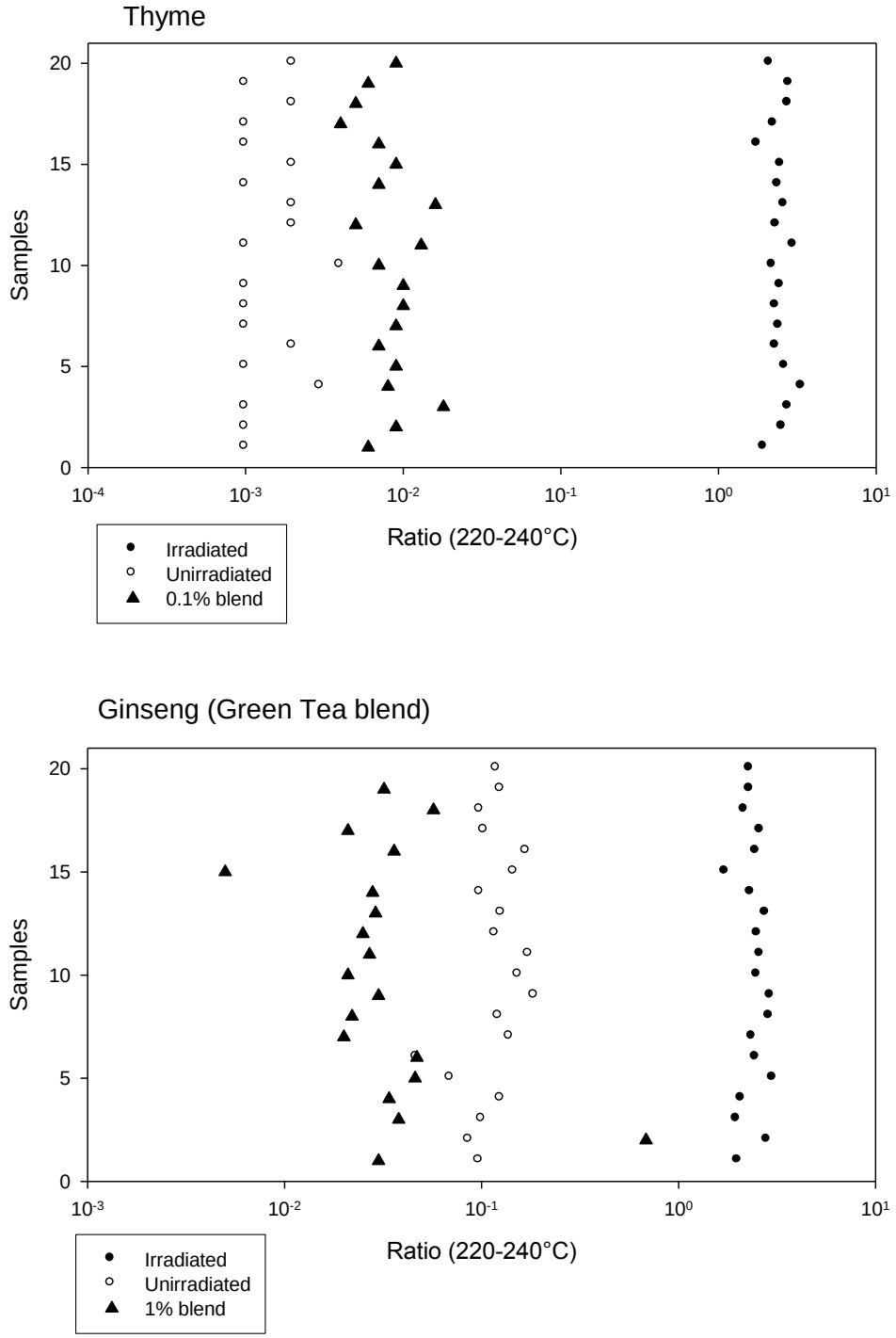


Figure 5.6 TL homogeneity testing glow ratio plot for thyme and ginseng (green tea)

5.3 Participants' Results

5.3.1 Quantitative results

TL results were returned by 16 of the 19 laboratories which received samples. 7 of these elected to analyse all 18 products; 7 opted for the reduced set of 9. The remaining 2 laboratories did not return data for all the samples they had chosen. The reduced set consisted of one herb (thyme), one spice (cumin) and one dietary supplement (guarana), presented in each of the three categories. TL participants were asked to return, first and second glow data, glow ratio, comments on glow curve shape and classification on two aliquots per sample; in some cases a different number of aliquots was reported.

Lab	TL reader	MDL	
1	Harshaw M 3500	0.13412634 nC	0.13412634 nC
2	Risö TL/OSL-DA15	499	
3	Harshaw 3500	0,620 nC	
4	Harshaw TLD-Reader 3500	20 nC	
6	Risoe TL-DA-15 Series No 194-05/2005-b	1693	
7	TLD 3500	3,12nC	
8	TLD 3500	0.899 nC	
9	Harshaw QS 3500	1.63 nC	
11	Riso TL/OSL	489	
12	Harshaw 3500	0.0582 nC	
13	TL-DA-10	177	
14	Harshaw 3500	0.25 nC	
15	Harshaw 3500	0.25 nC	
16	TL/OSL DA-20	1819 nC	
17	Harshaw TLD 3500	0.4405 nC	
18	RISÆ TL/OSL System, model TL-DA-15; TLF programme: 50-500°C; 6 °C/s	250.8	
19	Harshaw TLD 4000	0.53	

Table 5.1 Participants' instrumentation and MDL

Figures 5.6-5.9 present glow ratios for each laboratory and each product, with the 3 irradiation categories given different symbols. Intensities could not be plotted analogously to Figures 5.1-5.3 because participants used a variety of units for their measurements.

Tables 5.2 and 5.3 show participants' z-scores by laboratory and product respectively; these data are shown graphically in figures 5.10-5.12 (by laboratory) and figures 5.13-15 (by sample). Contour plots have not been produced because the variable number of data returned by each laboratory made them unsuitable.

From the figures it can be seen that participants' data distinguish the irradiated samples from the unirradiated ones, but the blends are not well separated. Some unirradiated materials produced extremely low glow ratios which have not been plotted. Separation by category is less apparent than for the homogeneity testing, but that featured 20 data points for each product, obtained on a single instrument. Some of the scatter in the participants' data may be explained by the use of different instruments with several different types of equipment with different irradiation sources, but there is also likely to be a component associated with

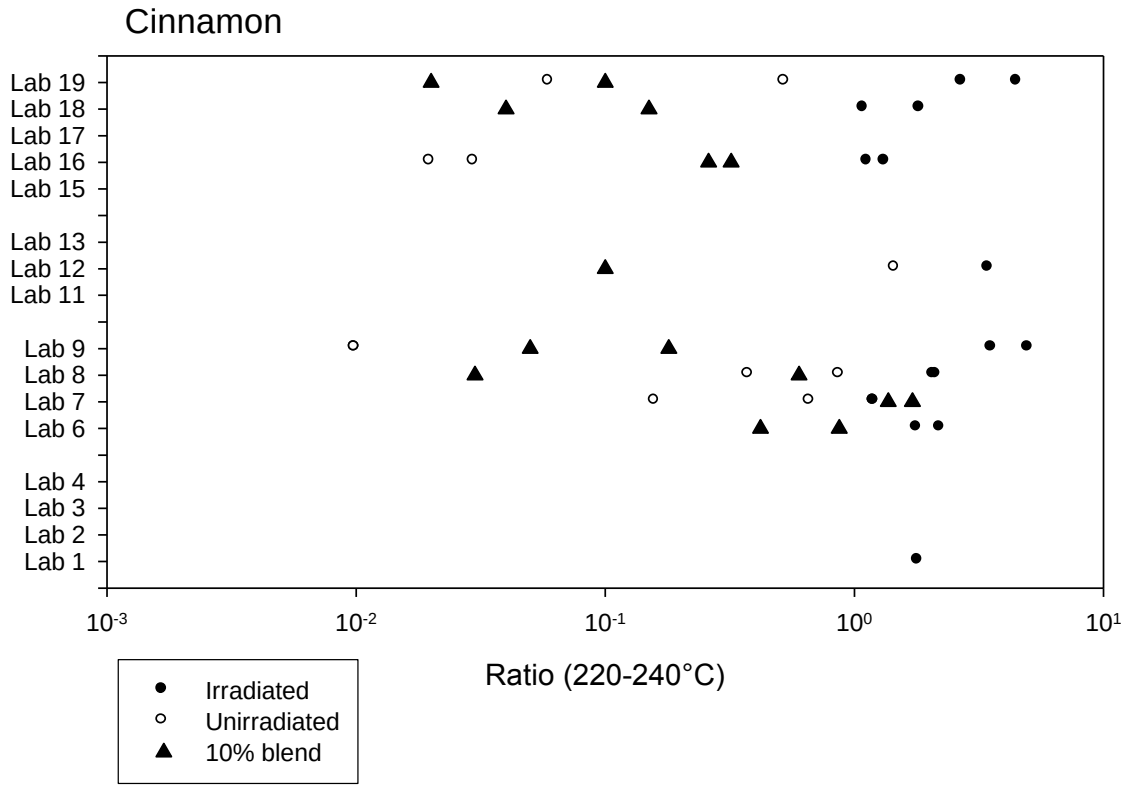
efficiency of mineral separation and sample heterogeneity and sensitivity may also be important.

Figures 5.10-12 show clearly that some laboratories have more closely spaced *z*-scores than others. The spread of *z*-scores is similar for each category, although the blends have a slightly tighter distribution with fewer elevated scores.

Plots of *z*-score by sample (Figures 5.13-5.15) show tails towards higher scores. It is not clear whether this is a result of properties of the materials, or caused by aspects of technique during the analyses.

The small number of samples, further reduced by some participants opting to perform fewer analyses and by the incomplete returns from some laboratories, has affected the statistics and may have limited their usefulness. It may be necessary to increase the number of samples if the PT scheme develops into a routine technique although this might deter some participants.

This report also includes a preliminary investigation into signal to background variation. Figures 5.16 displays this ratio for each product in each of the 3 categories, for the reference data and participants' results. It can be seen that the participants' data have a broadly similar distribution to the reference data. Since these data respond to instrumental sensitivity, efficiency of mineral separation and maintenance of low blank performance, they may provide a means of assessing overall laboratory performance. Plots for each separate laboratory could be constructed to this end.



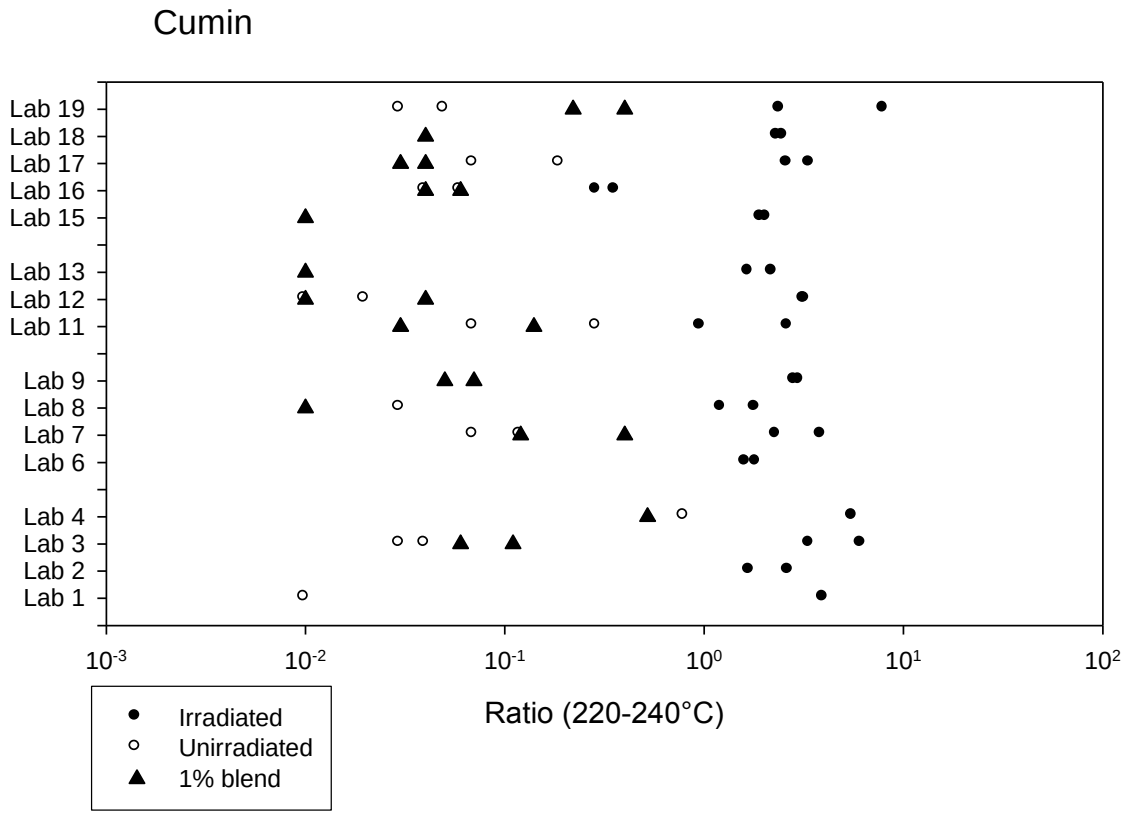
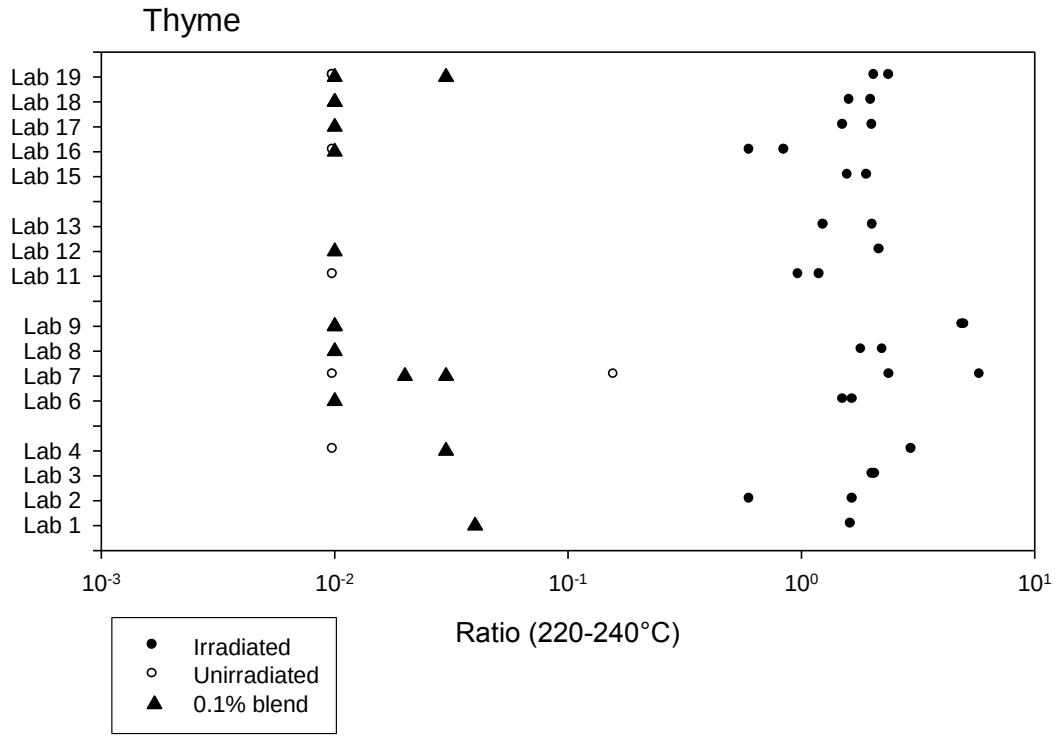


Figure 5.7 Participants' glow ratios for cinnamon and cumin



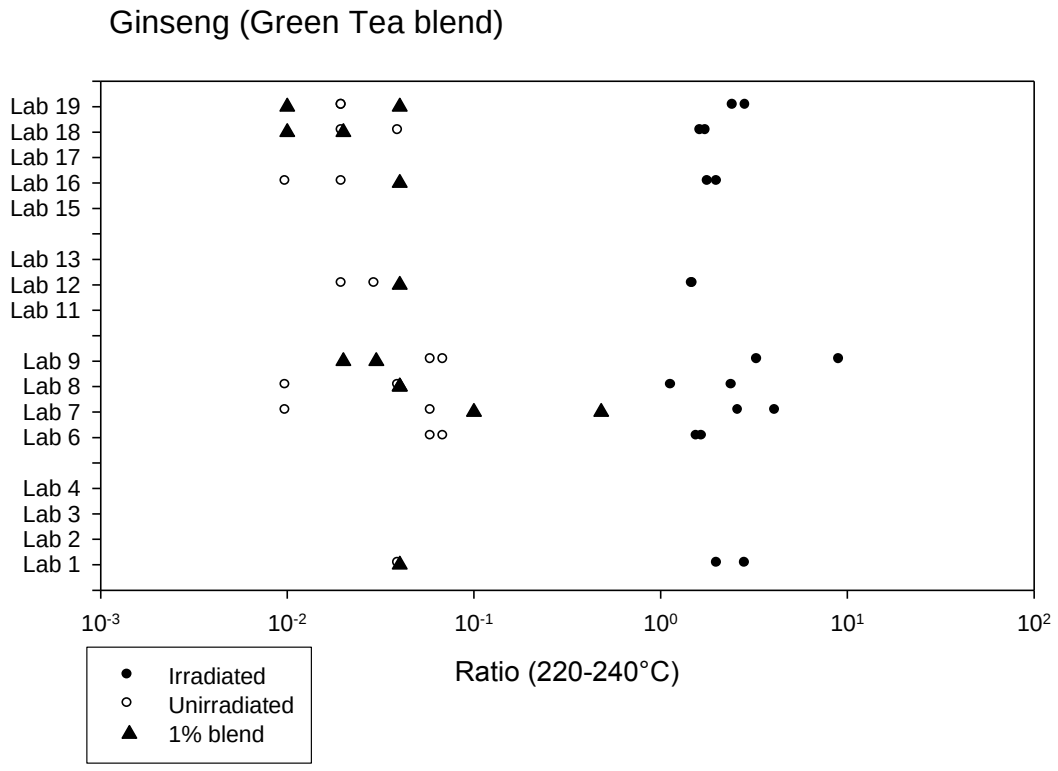
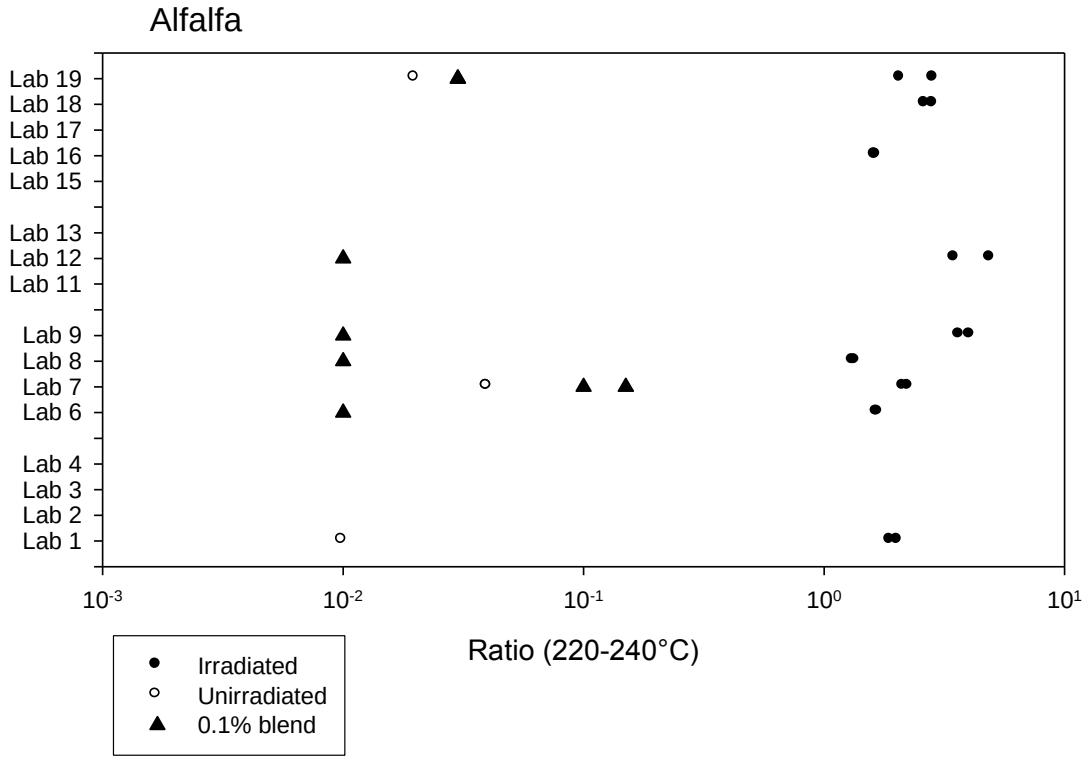


Figure 5.8 Participants' glow ratios for thyme and ginseng



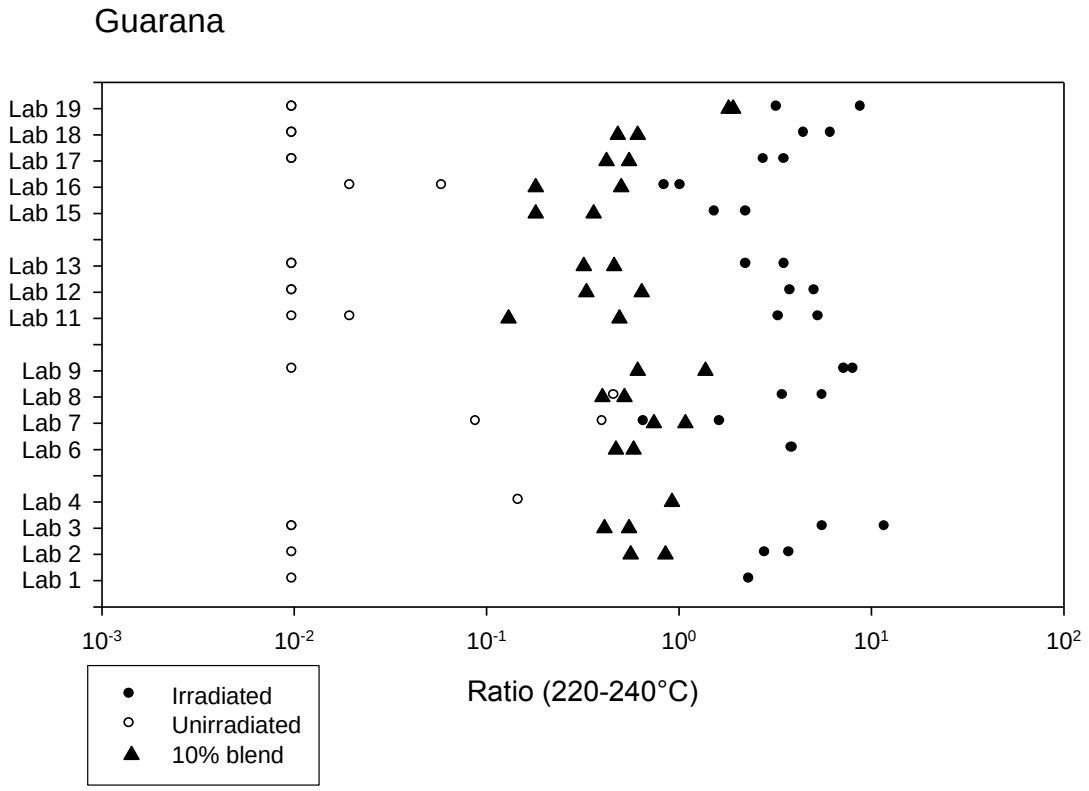


Figure 5.9 Participants' glow ratios for alfalfa and guarana

Description	Status	Lab 1	Lab 2	Lab 3	Lab 4	Lab 6	Lab 7	Lab 8	Lab 9	Lab 11	Lab 12	Lab 13	Lab 15	Lab 16	Lab 17	Lab 18	Lab 19
Cinnamon	U					(-)	5.56	34.72	-0.62		58.46			0.07		-0.82	20.38
						(-)	26.30	14.46	-0.54					0.42		-0.67	1.55
Cumin	U	-0.46	-0.75	0.22	20.11	(-)	1.14	-0.70	-0.73	1.02	-0.41	-0.75	-0.71	0.89	4.26	-0.74	-0.05
			-0.74	0.03		(-)	2.27	-0.07	-0.73	6.82	-0.21	-0.74	-0.65	0.40	1.17	-0.75	0.55
Thyme	U	0.56	-0.67	-1.54	9.05	(-)	194.62	0.72	-0.67	9.03	-1.31	0.36	-0.66	4.48	-0.26	-1.46	4.18
			-1.15	-1.58		(-)	6.26	-0.83	-0.67	-0.12		0.28	-1.51	-0.78	4.12	-1.18	13.87
Ginseng	U	-2.32				-1.82	-3.29	-2.44	-1.79		-2.71			-3.15		-2.94	-2.93
						-1.59	-1.62	-3.11	-1.59		-2.97			-2.96		-2.22	-2.88
Alfalfa	U	3.86				(-)	20.77	-0.71	-0.67		0.07			-1.49		-1.37	9.19
						(-)	21.14	-1.45	-1.25					-1.51		-1.33	-0.09
Guarana	U	-0.66	-0.70	-0.56	6.18	(-)	18.73	21.90	-0.80	-0.07	-0.72	-0.39	-0.87	1.74	-0.51	-0.69	-0.56
			-0.85	-0.71		(-)	3.54	-0.87	-0.56	-0.29	-0.70	-0.29	-0.84	-0.13	-0.41	-0.68	-0.66
Cinnamon	I	-0.33				-0.35	-0.95	0.01	2.92		1.36			-0.81		-0.30	0.59
						0.09	-0.94	-0.05	1.47					-1.02		-1.06	2.42
Cumin	I	1.58	-1.08	4.11	3.43	-0.93	-0.37	-0.96	0.27	0.02	0.63	-0.50	-0.66	-2.64	-0.01	-0.16	6.24
			0.04	0.89		-1.18	1.45	-1.64	0.44	-1.94	0.69	-1.11	-0.81	-2.73	0.90	-0.33	-0.26
Thyme	I	-2.26	-5.11	-1.19	1.43	-2.59	-0.17	-0.60	6.74	-4.08	-0.79	-3.31	-1.46	-4.42	-2.60	-1.26	-0.20
			-2.18	-1.03		-2.18	9.29	-1.76	7.04	-3.46		-1.17	-2.41	-5.11	-1.21	-2.32	-1.10
Ginseng	I	-1.18				-2.49	0.58	-0.01	2.60		-2.69			-1.18		-2.26	1.30
		1.23				-2.18	4.97	-3.73	19.43		-2.76			-1.80		-1.94	0.09
Alfalfa	I	-1.21				-2.19	0.37	-3.51	6.51		5.77			-2.23		2.03	-0.38
		-0.61				-2.10	-0.12	-3.66	8.21		11.94			-2.33		2.95	2.98
Guarana	I	-1.65	-1.39	3.37		-0.83	-2.01	-1.03	0.98	-1.12	-0.86	-1.00	-1.69	-2.44	-1.41	-0.51	1.81
			-0.89	0.09		-0.80	-2.53	0.09	1.41	-0.05	-0.18	-1.70	-2.06	-2.34	-1.00	0.40	-1.16
Cinnamon	B					-0.17	1.39	-0.63	-0.46		-0.56			-0.28		-0.50	-0.55
						0.38	0.97	0.05	-0.62					-0.36		-0.62	-0.65
Cumin	B		-0.50	-0.16	2.40	(-)	0.17	-0.49	-0.26	-0.36	-0.48	-0.47	-0.51	-0.31	-0.35	-0.27	1.71
			-0.52	0.10		(-)	1.70	-0.50	-0.14	0.24	-0.30	-0.52	-0.45	-0.19	-0.29	-0.50	0.71
Thyme	B	9.68	-2.32	-1.39	5.29	-1.61	2.91	-0.32	0.37	-1.39	-0.99	-1.17	-1.69	-0.21	0.39	-0.59	7.15
			-2.32	-1.11		-1.04	7.34	-2.22	-1.04	-1.81		-2.43	-2.25	-2.15		-0.20	0.93
Alfalfa	B	-0.27				-0.24	1.23	-0.17	-0.27		-0.19			-0.32		-0.31	0.07
						-0.27	2.14	-0.32	-0.22					-0.30		-0.30	0.12
Green tea	B	-0.16				(-)	0.28	-0.16	-0.28		-0.18			-0.13		-0.32	-0.17
						(-)	2.81	-0.15	-0.22					-0.39		-0.32	-0.37
Guarana	B		-0.10	-0.74	1.44	-0.01	0.68	-0.28	0.12	-1.93	0.24	-1.10	-1.70	-0.33	-0.67	0.13	5.61
			1.13	-0.15		-0.48	2.10	-0.78	3.34	-0.39	-1.06	-0.51	-0.95	-1.71	-0.15	-0.45	5.19

Table 5.2 Participants' *z*-score for TL analyses by laboratory

Descript	Cin	Cumin	Thy	Gin	Alfa	Gua	Cin	Cumin	Thy	Gin	Alfa	Gua	Cin	Cumin	Thy	Alfa	GT	Gua
Status	U	U	U	U	U	U	I	I	I	I	I	I	B	B	B	B	B	B
Lab 1		-0.46	0.56	-2.32	3.86	-0.66	-0.33	1.58	-2.26	-1.18	-1.21	-1.65			9.68	-0.27	-0.16	
										1.23	-0.61							
Lab 2		-0.75	-0.67			-0.70		-1.08	-5.11			-1.39		-0.50	-2.32			-0.10
		-0.74	-1.15			-0.85		0.04	-2.18			-0.89		-0.52	-2.32			1.13
Lab 3		0.22	-1.54			-0.56		4.11	-1.19			3.37		-0.16	-1.39			-0.74
		0.03	-1.58			-0.71		0.89	-1.03			0.09		0.10	-1.11			-0.15
Lab 4		20.11	9.05			6.18		3.43	1.43					2.40	5.29			1.44
Lab 6	(-)	(-)	(-)	-1.82	(-)	(-)	-0.35	-0.93	-2.59	-2.49	-2.19	-0.83	-0.17	(-)	-1.61	-0.24	(-)	-0.01
	(-)	(-)	(-)	-1.59	(-)	(-)	0.09	-1.18	-2.18	-2.18	-2.10	-0.80	0.38	(-)	-1.04	-0.27	(-)	-0.48
Lab 7	5.56	1.14	194.62	-3.29	20.77	18.73	-0.95	-0.37	-0.17	0.58	0.37	-2.01	1.39	0.17	2.91	1.23	0.28	0.68
	26.30	2.27	6.26	-1.62	21.14	3.54	-0.94	1.45	9.29	4.97	-0.12	-2.53	0.97	1.70	7.34	2.14	2.81	2.10
Lab 8	34.72	-0.70	0.72	-2.44	-0.71	21.90	0.01	-0.96	-0.60	-0.01	-3.51	-1.03	-0.63	-0.49	-0.32	-0.17	-0.16	-0.28
	14.46	-0.07	-0.83	-3.11	-1.45	-0.87	-0.05	-1.64	-1.76	-3.73	-3.66	0.09	0.05	-0.50	-2.22	-0.32	-0.15	-0.78
Lab 9	-0.62	-0.73	-0.67	-1.79	-0.67	-0.80	2.92	0.27	6.74	2.60	6.51	0.98	-0.46	-0.26	0.37	-0.27	-0.28	0.12
	-0.54	-0.73	-0.67	-1.59	-1.25	-0.56	1.47	0.44	7.04	19.43	8.21	1.41	-0.62	-0.14	-1.04	-0.22	-0.22	3.34
Lab 11		1.02	9.03			-0.07		0.02	-4.08			-1.12		-0.36	-1.39			-1.93
		6.82	-0.12			-0.29		-1.94	-3.46			-0.05		0.24	-1.81			-0.39
Lab 12	58.46	-0.41	-1.31	-2.71	0.07	-0.72	1.36	0.63	-0.79	-2.69	5.77	-0.86	-0.56	-0.48	-0.99	-0.19	-0.18	0.24
		-0.21		-2.97		-0.70		0.69		-2.76	11.94	-0.18		-0.30				-1.06
Lab 13		-0.75	0.36			-0.39		-0.50	-3.31			-1.00		-0.47	-1.17			-1.10
		-0.74	0.28			-0.29		-1.11	-1.17			-1.70		-0.52	-2.43			-0.51
Lab 15		-0.71	-0.66			-0.87		-0.66	-1.46			-1.69		-0.51	-1.69			-1.70
		-0.65	-1.51			-0.84		-0.81	-2.41			-2.06		-0.45	-2.25			-0.95
Lab 16	0.07	0.89	4.48	-3.15	-1.49	1.74	-0.81	-2.64	-4.42	-1.18	-2.23	-2.44	-0.28	-0.31	-0.21	-0.32	-0.13	-0.33
	0.42	0.40	-0.78	-2.96	-1.51	-0.13	-1.02	-2.73	-5.11	-1.80	-2.33	-2.34	-0.36	-0.19	-2.15	-0.30	-0.39	-1.71
Lab 17		4.26	-0.26			-0.51		-0.01	-2.60			-1.41		-0.35	0.39			-0.67
		1.17	4.12			-0.41		0.90	-1.21			-1.00		-0.29				-0.15
Lab 18	-0.82	-0.74	-1.46	-2.94	-1.37	-0.69	-0.30	-0.16	-1.26	-2.26	2.03	-0.51	-0.50	-0.27	-0.59	-0.31	-0.32	0.13
	-0.67	-0.75	-1.18	-2.22	-1.33	-0.68	-1.06	-0.33	-2.32	-1.94	2.95	0.40	-0.62	-0.50	-0.20	-0.30	-0.32	-0.45
Lab 19	20.38	-0.05	4.18	-2.93	9.19	-0.56	0.59	6.24	-0.20	1.30	-0.38	1.81	-0.55	1.71	7.15	0.07	-0.17	5.61
	1.55	0.55	13.87	-2.88	-0.09	-0.66	2.42	-0.26	-1.10	0.09	2.98	-1.16	-0.65	0.71	0.93	0.12	-0.37	5.19

Table 5.3 Participants' *z*-score for TL analyses by sample

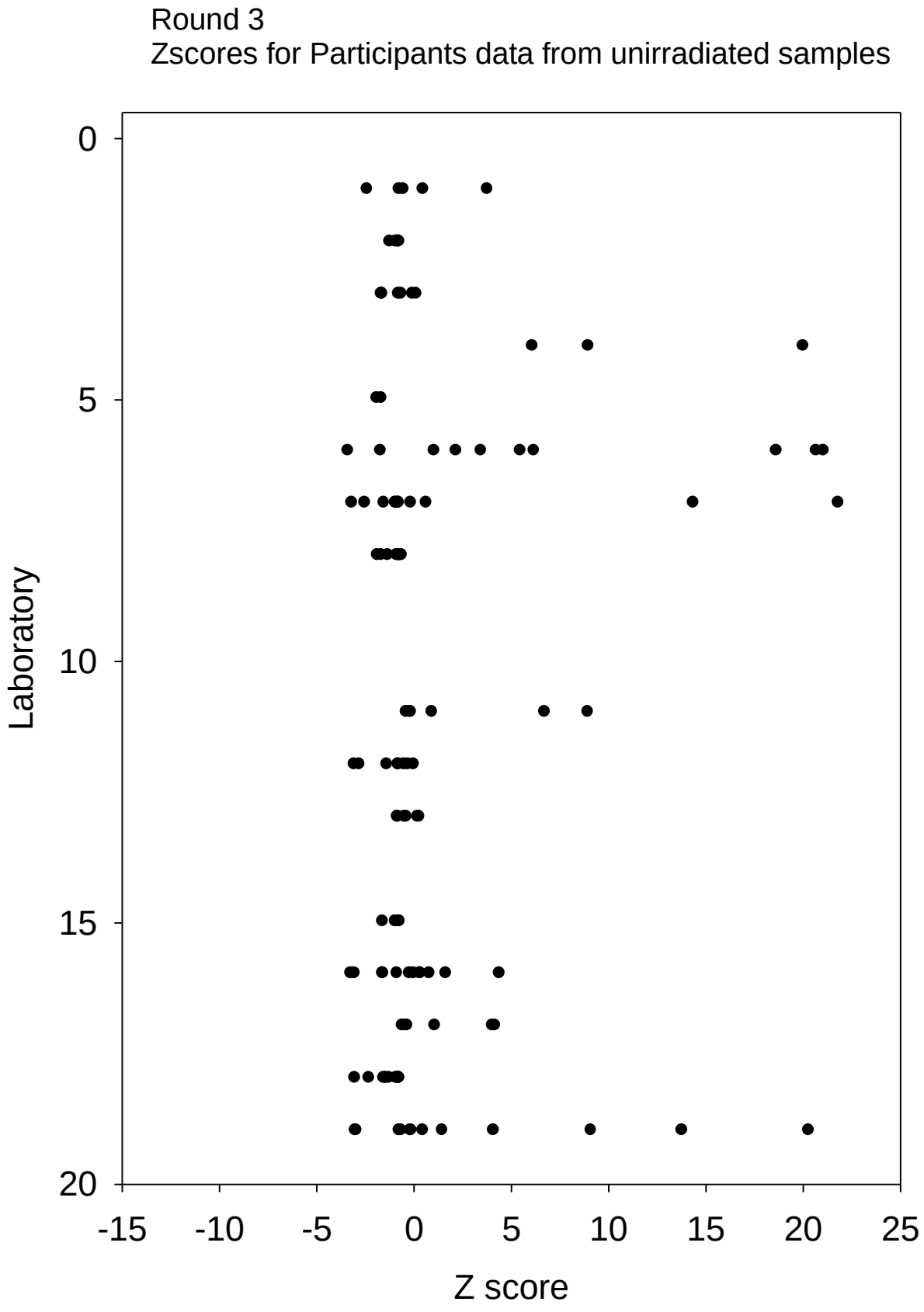


Figure 5.10 Participants' TL *z*-scores for unirradiated samples by laboratory

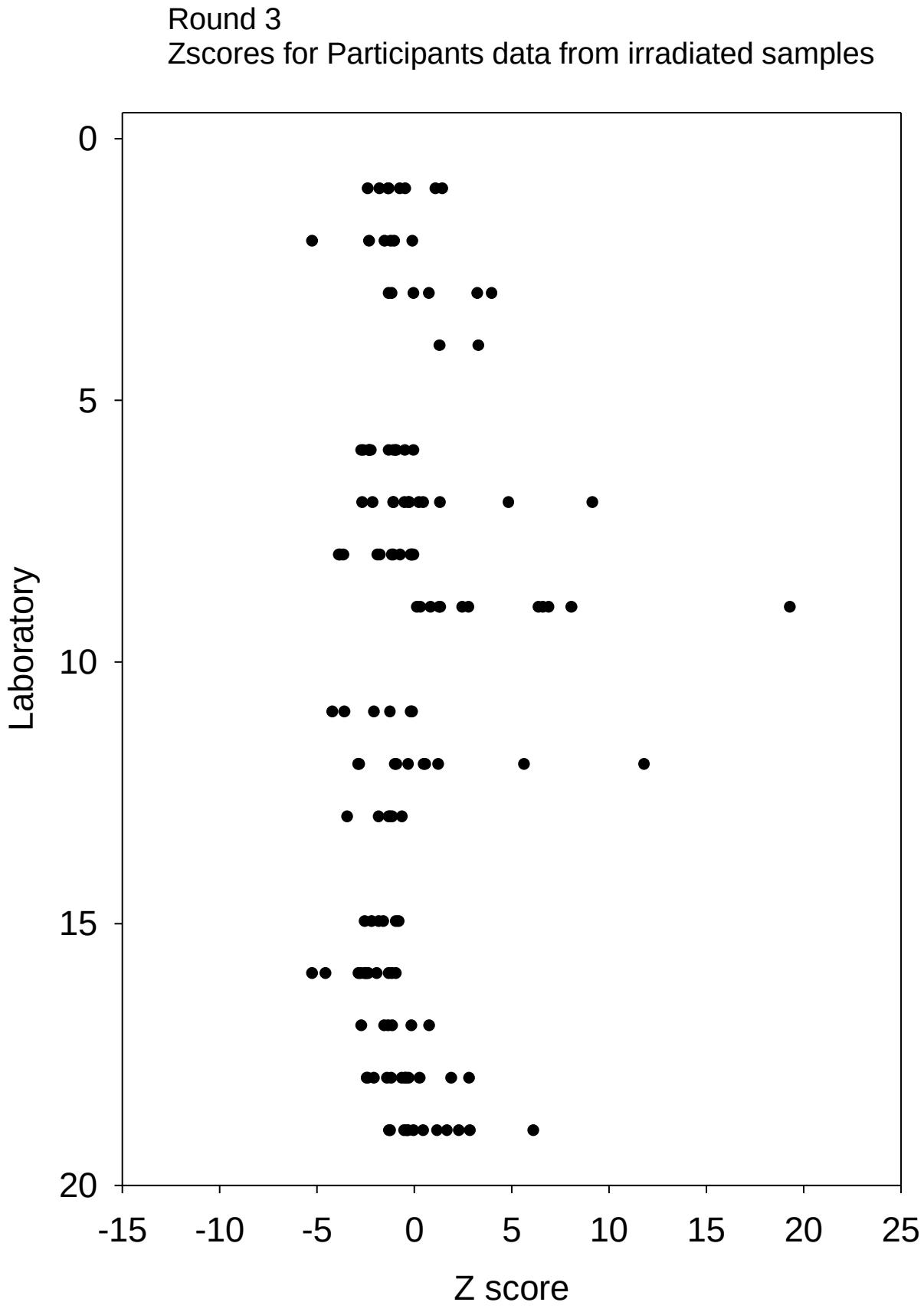


Figure 5.11 Participants' TL *z*-scores for irradiated samples by laboratory

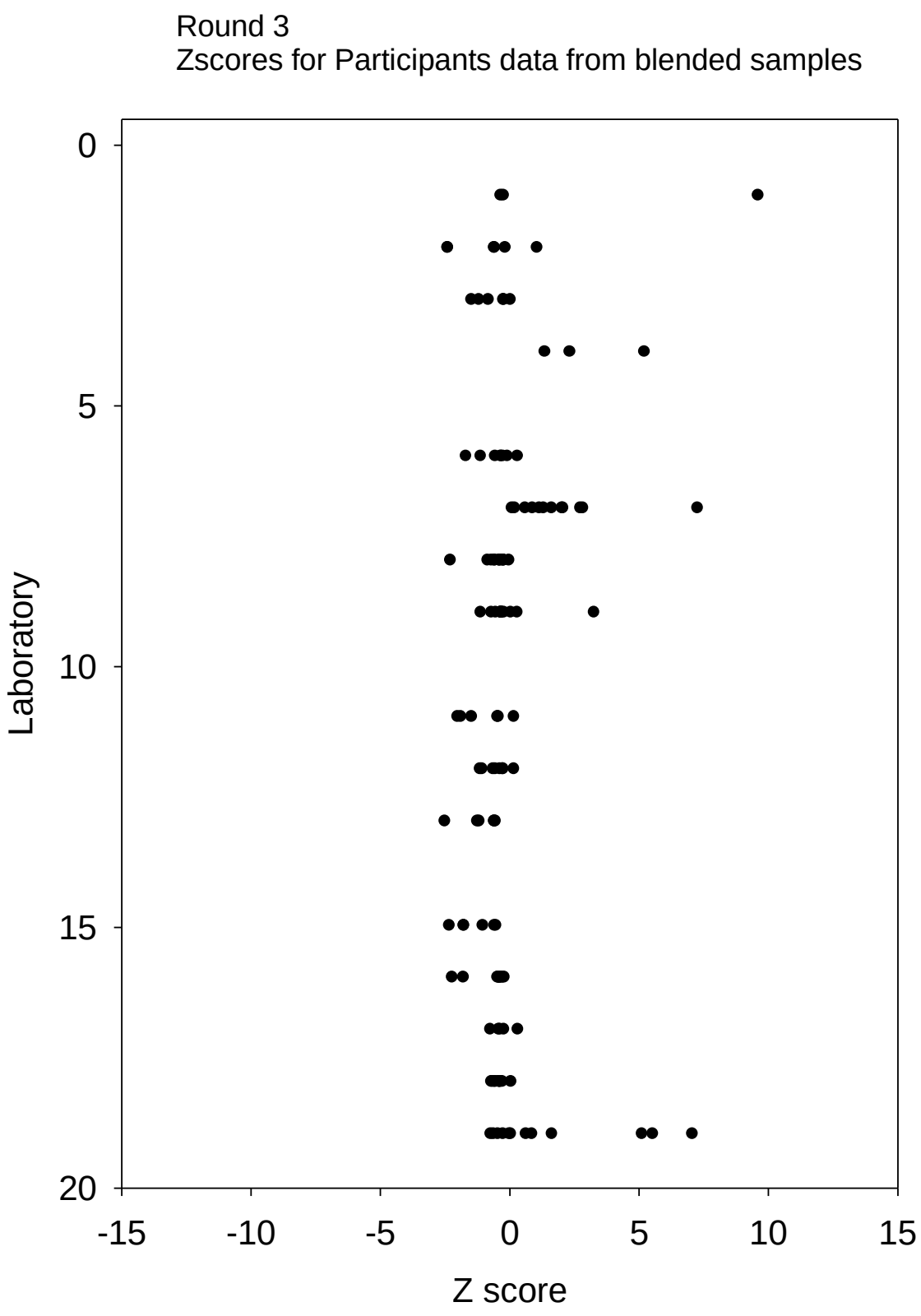


Figure 5.12 Participants' TL *z*-scores for blended samples by laboratory

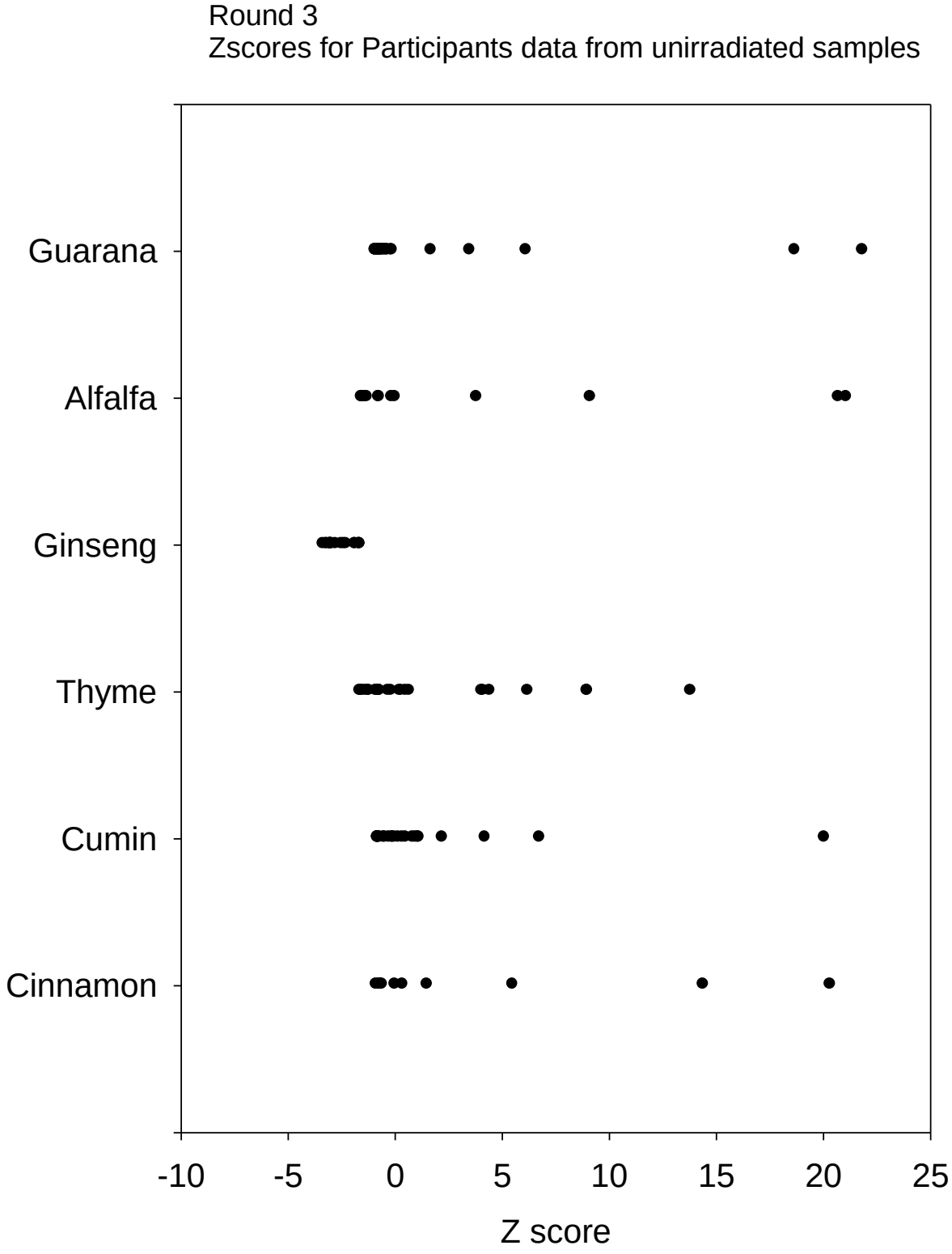


Figure 5.13 Participants' TL *z*-scores for unirradiated samples by sample

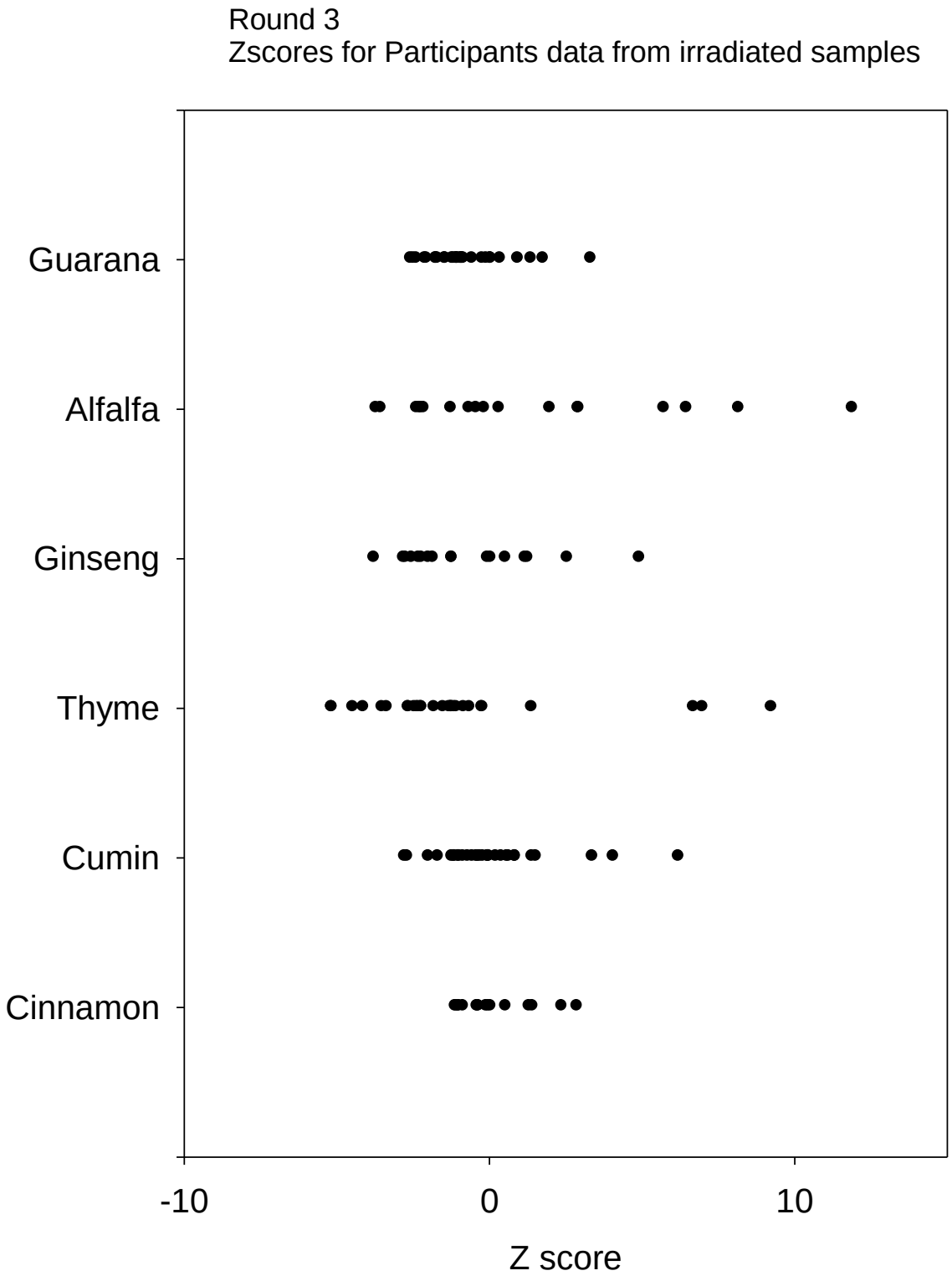


Figure 5.14 Participants' TL *z*-scores for irradiated samples by sample

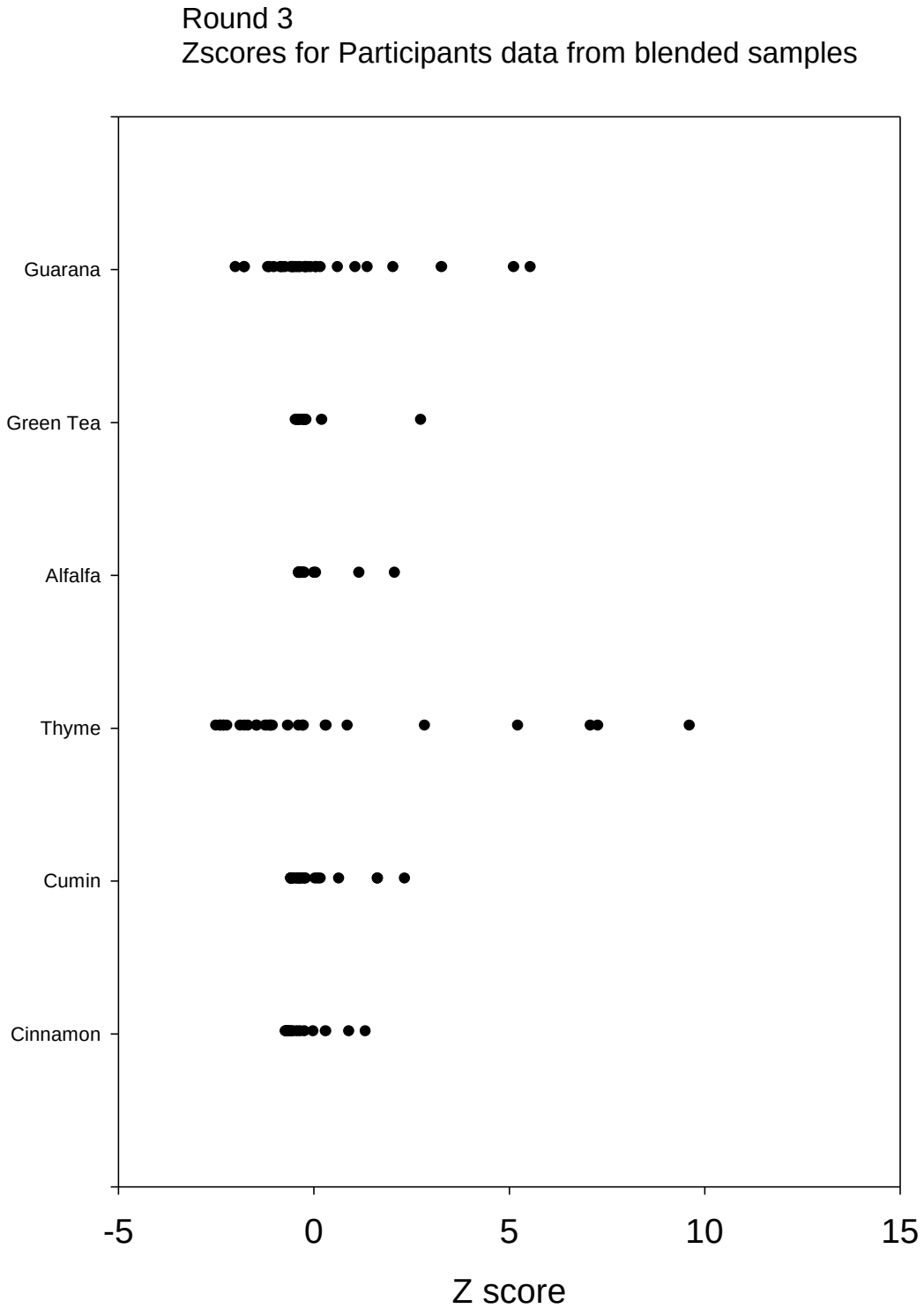


Figure 5.15 Participants' TL *z*-scores for blended samples by sample

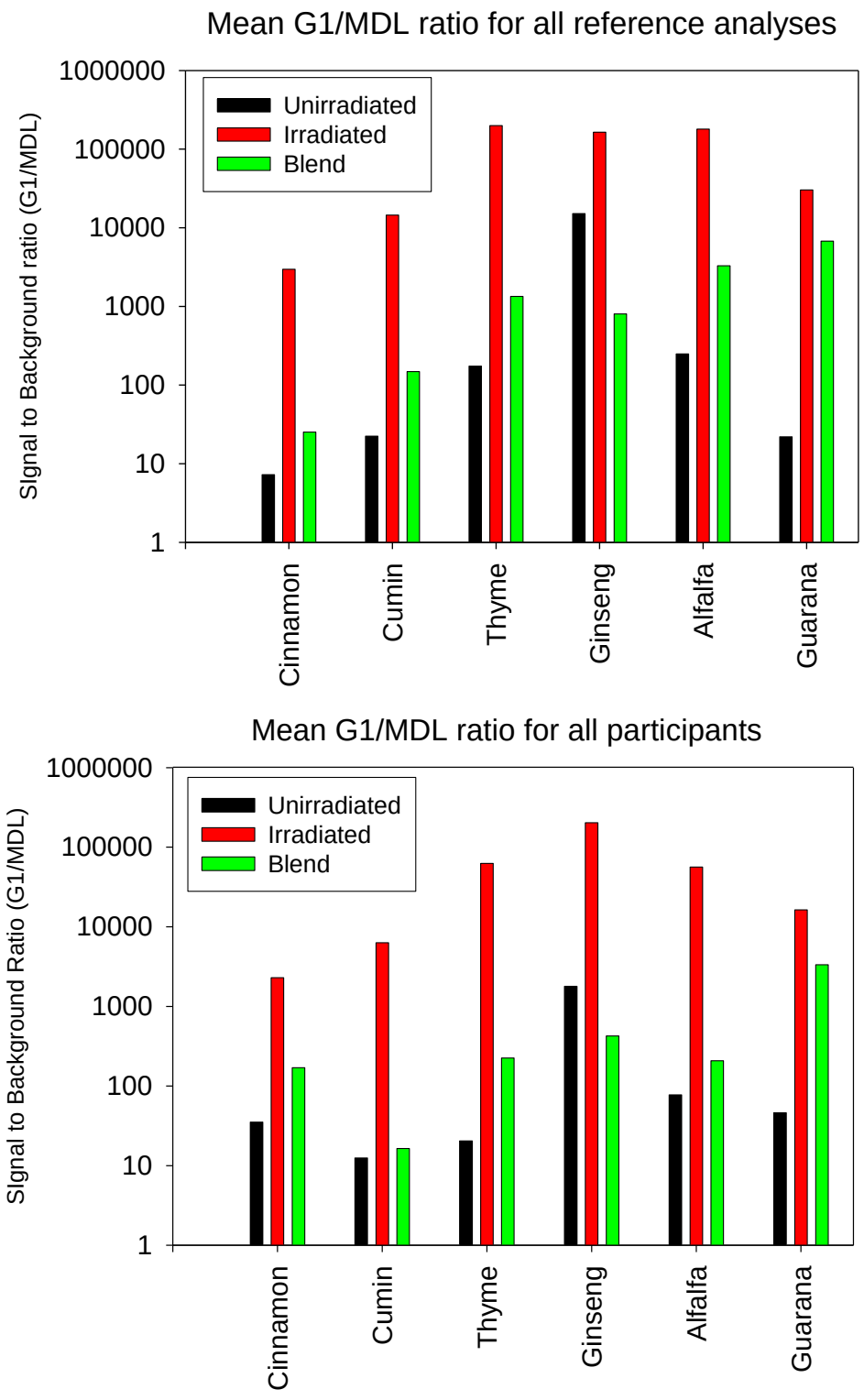


Figure 5.16 Mean signal to background ratios for reference analyses and participants data

5.3.2 Qualitative results

Participants were asked to return classifications of the samples as well as descriptions of the glow curves. These can both be used to assess whether they corrected identified the irradiation status of the materials. Tables 5.5and 5.6 detail their classifications compared with the known status of the material, by laboratory and by product respectively.

Evaluation of "correct" and "incorrect" results is based on the declared status of the samples as purchased, with the exception of the ginseng where the actual status of the product differed from what was declared. Reference analysis revealed this sample to be a mixture containing irradiated material, so participants who classified it as such were deemed to have identified it correctly. Two other products (unirradiated thyme and cumin) displayed small low temperature signals in the reference set, but only 2 participants reported similar results; identification as unirradiated was regarded as correct.

	All products			Unirradiated			Irradiated			Blends		
	Correct	Incorrect	% correct	Correct	Incorrect	% correct	Correct	Incorrect	% correct	Correct	Incorrect	% correct
Lab 1	7	7	50.00%	1	4	20.00%	6	0	100.00%	0	3	0.00%
Lab 2	6	3	66.67%	3	0	100.00%	3	0	100.00%	0	3	0.00%
Lab 3	8	1	88.89%	2	1	66.67%	3	0	100.00%	3	0	100.00%
Lab 4	7	2	77.78%	3	0	100.00%	3	0	100.00%	1	2	33.33%
Lab 6	14	4	77.78%	6	0	100.00%	6	0	100.00%	2	4	33.33%
Lab 7	11	7	61.11%	5	1	83.33%	6	0	100.00%	0	6	0.00%
Lab 8	15	3	83.33%	5	1	83.33%	6	0	100.00%	4	2	66.67%
Lab 9	17	1	94.44%	6	0	100.00%	6	0	100.00%	5	1	83.33%
Lab 11	7	2	77.78%	3	0	100.00%	3	0	100.00%	1	2	33.33%
Lab 12*	13	4	72.22%	5	1	83.33%	6	0	100.00%	2	3	33.33%
Lab 13	8	1	88.89%	3	0	100.00%	3	0	100.00%	2	1	66.67%
Lab 15	6	3	66.67%	2	1	66.67%	3	0	100.00%	1	2	33.33%
Lab 16	12	6	66.67%	6	0	100.00%	6	0	100.00%	0	6	0.00%
Lab 17	7	2	77.78%	2	1	66.67%	3	0	100.00%	2	1	66.67%
Lab 18	16	2	88.89%	6	0	100.00%	6	0	100.00%	4	2	66.67%
Lab 19**	10	6	55.56%	4	1	66.67%	6	0	100.00%	0	5	0.00%
Total	164	54		62	11		75	0		27	43	
Percentage	74.21%	24.43%		83.78%	14.86%		100.00%	0.00%		37.50%	59.72%	

*plus one indeterminate (blend)

**plus two indeterminate (1 blend 1 unirradiated)

Table 5.4 Participants' qualitative TL results by laboratory

	All products			Unirradiated			Irradiated			Blends		
	Correct	Incorrect	% correct	Correct	Incorrect	% correct	Correct	Incorrect	% correct	Correct	Incorrect	% correct
Cinnamon	18	6	75.00%	7	1	87.50%	9	0	100.00%	2	5	28.57%
Cumin	36	10	78.26%	14	2	87.50%	16	0	100.00%	6	8	42.86%
Thyme	38	10	79.17%	15	1	93.75%	16	0	100.00%	7	9	43.75%
Ginseng**	16	2	88.89%	7	2	77.78%	9	0	100.00%			
Alfalfa	18	8	69.23%	6	2	75.00%	9	0	100.00%	3	6	33.33%
Green Tea	4	5	44.44%							4	5	44.44%
Guarana	34	13	72.34%	13	3	81.25%	16	0	100.00%	5	10	33.33%
Total	164	54		62	11		75	0		27	43	
Percentage	74.21%	24.43%		84.93%	15.07%		100.00%	0.00%		38.57%	61.43%	

** this product, supplied as unirradiated, contains an irradiated component; qualitative results without this product are as below

	All products			Unirradiated			Irradiated			Blends		
	Correct	Incorrect	% correct	Correct	Incorrect	% correct	Correct	Incorrect	% correct	Correct	Incorrect	% correct
Cinnamon	18	6	75.00%	7	1	87.50%	9	0	100.00%	2	5	28.57%
Cumin	36	10	78.26%	14	2	87.50%	16	0	100.00%	6	8	42.86%
Thyme	38	10	79.17%	15	1	93.75%	16	0	100.00%	7	9	43.75%
Alfalfa	18	8	69.23%	6	2	75.00%	9	0	100.00%	3	6	33.33%
Green Tea	4	5	44.44%							4	5	44.44%
Guarana	34	13	72.34%	13	3	81.25%	16	0	100.00%	5	10	33.33%
Total	148	52		55	9		66	0		27	43	
Percentage	74.00%	26.00%		85.94%	14.06%		100.00%	0.00%		38.57%	61.43%	

Table 5.5 Participants' qualitative TL results by sample

For Round 3, where not all participants analysed all samples, a direct comparison with "correct" percentages from Round 2 should be treated with caution. There does not appear to be a correlation between number of samples analysed and performance; laboratories 3, 8,9 and 18 did particularly well in terms of overall percentage correct. Two of these laboratories (9 and 18) analysed all 18 products. Laboratory 19, which did less well, had most difficulty identifying the blends as such; this may be an indication that they were using less sensitive equipment. This is supported by their being unable to evaluate two of the samples.

Lab 1, the least successful, returned an unusual set of results, with one aliquot analysed for each of 12 samples, and 2 each for a further 2 which does not correspond with either of the options in the Protocol. Their evaluation is also at variance with the ratios and peak shapes detailed in their response. Their comments describe 6 samples as "negative in water" although in all cases these samples are evaluated as positive. For the purposes of this report, their results have been included without any attempt at untangling them.

Lab 4 failed to obtain a second aliquot for any of the samples, suggesting a deviation from the method of preparing 2 aliquots in parallel from each sample. Reference analysis produced adequate yield from all materials, and plentiful yield from some, so participants should have been able to, and in general did, extract enough for 2 aliquots of at least some of the samples even if separation into 2 portions only took place at the dispensing stage.

Lab 12 returned results from at least one aliquot of each sample, and had a success rate of 72% (with one sample not producing a determination), despite apparently analysing one aliquot of some of the samples and 3 or 4 of each of the others. No reason was given for not performing duplicate analysis on all products.

Overall performance is 74% correct, with correct identification in 86% and 100% of unirradiated and irradiated samples respectively, based on those cases where an evaluation was returned. As before, the blends presented more of a challenge, with a minority of them identified as containing irradiated material. In Round 2, calculated percentages included "null" responses. For Round 3, due to the different number of samples analysed by different laboratories, percentages were calculated only for returned results.

6. DISCUSSION AND CONCLUSIONS

As shown in the previous sections the third round of the trial was successfully implemented, with test material procurement, preparation and distribution, homogeneity testing and the second measurement round taking place within a few months.

The PSL reference data sets obtained in the organising laboratory resemble earlier published data from the original PSL research and inter-laboratory trial data sets, as does TL homogeneity testing.

PSL screening results from 35 participating laboratories were returned in a timely manner and generally show equivalence to the SUERC reference data. New laboratories who had not participated in the earlier rounds performed as well as those who had.

The data sets have been explored using descriptive statistics and in tabular and graphical forms. There is still evidence of additional inter-laboratory variation in comparison with the reference set. Comparison of qualitative outcomes between the rounds indicates stability of performance, even with different materials and a larger number of new participants than occurred between the first two rounds. In Round 2 in was concluded that it might be possible to develop a design for a "sparse" study utilising smaller numbers of samples, and thus capable of being sustained beyond the timescale of this developmental project. Round 3 simulates this and has overall proved to be a success, although there is some evidence that reducing the number of samples adversely affects statistical examination of the data. In particular, the comparison of pooled *z*-scores with percentage qualitatively "correct" determinations suffers from the inevitable quantization of the percentages, which is more obvious with fewer samples.

Calibrated PSL measurements were undertaken a successfully by 10 laboratories using a diverse set of irradiation facilities similar to Round 2. The results are again broadly comparable to the reference analyses, with good discrimination between irradiated and unirradiated samples in most cases, and blends being distributed in the Calibrated PSL plots in the loci between unirradiated and irradiated samples. Also as in Round 2 the proportion of samples with very low sensitivity, where Calibrated PSL measurements can very usefully call into question the status of negative screening results, was extremely low. The absolute number of such samples was also affected by reducing the total number of samples presented. Therefore it has not been possible to demonstrate that calibration had a large impact on evaluation from screening, possibly reflected in the paucity of participants' comments on this.

For the TL study this round included 18 samples from 6 products, in unirradiated, irradiated and blended forms. As indicated above, green tea was substituted for ginseng in the blend, making a total of 7 products used. The reference analyses showed that it was possible to recover minerals from all products; the data sets showed a high degree of internal consistency in separating irradiated and unirradiated examples although the sensitivities of the newly acquired materials and the concentrations chosen for the blends meant that the concentration ordered sequence seen in round 2 was no longer apparent. Reference analyses comprised 20 preparations per product producing 360 individual TL determinations, equivalent to 180 conventional duplicated TL analyses. Sixteen participants returned data for a variable number of products and overall performance was good, with some inter-laboratory variation in successful identifications. Blends again presented more difficulties, and irradiated samples, identified 100% correctly, remain the easiest. The option of using a reduced set of samples does not appear to have led to a higher percentage of correct determinations for those laboratories using the smaller set; for a routine proficiency test it might be more appropriate to use the full set.

The use of *z*-scores for TL requires further examination; this round also introduced signal to background ratio as a means of assessing overall performance. The two statistics together could perhaps form the basis of a certificate of proficiency during commercial application of the scheme.

The third round of proficiency testing analysis under this project has again resulted in a high return rate of extremely good and useful data from laboratories engaged in PSL and TL analysis of irradiated foods. Participation in such work represents an important commitment by participants and by the study organisers to the ongoing task of ensuring high analytical quality in routine determinations. It is to be hoped that this work will lead to sustained activities in support of food irradiation analyses.

APPENDIX A: TEST SAMPLE DISTRIBUTION AND STUDY PROTOCOL

Food Standards Agency Sponsored Development of Proficiency Testing for Detection of Irradiated Foods. Round 3 (PSL and TL)

3. 1. ROUND 3

The third round of the Food Standards Agency project to develop PT schemes for detection of irradiated foods is now underway. Many thanks for your continued participation in this project. Round 3 involves both PSL measurements and TL measurements. The data returns should be made electronically using the EXCEL spreadsheets issued. Please enter your **lab number as marked on the samples** (it may be different for the PSL and TL parts) in the data sheets.

The protocols for PSL and TL analysis are summarised here.

4. 2. PSL ANALYSIS

All PSL participants are asked to return screening results from round 3, as outlined below in section 2.1. Some participants have also indicated a desire to continue to perform Calibrated PSL measurements as well; this is outlined in section 2.2.

This time you will receive a new sample of irradiated paprika standard material, plus a set of 18 numbered samples for PSL analysis, and some petri dishes.

2.1 PSL Screening

(i) Please set your system up in your normal manner, ready for use in accordance with EN13751.

(ii) Before working on the test samples please dispense and measure **10 different portions of the new irradiated paprika standard**. This is to provide us with a measurement of the sensitivity of your instrument to this new material, and does not form part of the blind trial. For round 3, rather than dispensing these standard samples by volume, please weigh **2 g**. of the standard into the petri dish, shake or spread it to cover the base, and record the counting data. The material may be discarded after measurement. If you wish to re-use the sample petri dish for all 10 paprika measurements you may. But please be careful not to spread irradiated paprika to the outsides of the dishes or to the instrument chamber.

(iii) Then measure the samples using a new pair of petri dishes for each of the numbered test samples. You should dispense and measure these in the numbered sequence using your normal procedure.

Measure each sample in duplicate, and record the PSL screening results in the EXCEL spreadsheet provided by disk and email.

Please be careful when dispensing, handling and measuring the samples to avoid cross-contamination, or contamination of the sample chamber. Some of the samples are irradiated, and may have high sensitivities.

PSL screening results should be returned to the organisers by email (to D.Sanderson@suerc.gla.ac.uk &/or L.Carmichael@suerc.gla.ac.uk) both using the EXCEL spreadsheet and also with a copy of the PSL summary files for the runs used.

2.2 Calibrated PSL

For those laboratories continuing with Calibrated PSL measurements, seal your petri dishes and bag them after screening measurements. They should then be irradiated using a gamma or electron beam facility to a dose of 1 kGy. Then after a suitable post-irradiation delay (at least 24 hours) they should be re-measured and evaluated.

Return these results also using the combination of EXCEL spreadsheet and back-up PSL summary file.

3. TL laboratories

Participating TL laboratories are supplied with a set of 18 numbered test samples. **Those laboratories who indicated that they would prefer to analyse only 9 samples should only do samples:**

2, 3, 6, 8, 12, 14, 15, 16, 18

The test samples should be prepared in duplicate following EN1788 compatible procedures, including appropriate quality control measures (particularly blanks). Prepared samples should then be read out by TL (typically from 0-400°C at 5°C s^{-1}) to produce **first glow** TL data. The samples should then be irradiated using a ^{60}Co, ^{90}Sr or equivalent radiation course to a dose of 1 kGy, stored or preheated (eg at 50°C for one hour) and then remeasured to obtain **second glow** TL data. Return data for each sample comprising the first glow and second glow intensities, the TL glow ratio, observations on glow1 peak shapes and your evaluation. The samples may be irradiated, untreated, or mixtures containing irradiated materials. Please evaluate your data in these terms.

The TL results should be returned both in the supplied EXCEL spreadsheet (in the TL results page)

4. Return dates

Please return **PSL screening results, Calibrated PSL** and **TL results no later than 14th May 2007**.

APPENDIX B – RAW DATA FOR REFERENCE MATERIALS

Table B.1 Raw data for unirradiated test material – screening PSL using DOS system (cinnamon and cumin)

Product	Pot	Aliquot	Terminal count
Cinnamon	1	1	300
Cinnamon	1	2	371
Cinnamon	2	1	232
Cinnamon	2	2	241
Cinnamon	3	1	319
Cinnamon	3	2	352
Cinnamon	4	1	269
Cinnamon	4	2	359
Cinnamon	5	1	304
Cinnamon	5	2	314
Cinnamon	6	1	276
Cinnamon	6	2	166
Cinnamon	7	1	282
Cinnamon	7	2	531
Cinnamon	8	1	364
Cinnamon	8	2	257
Cinnamon	9	1	324
Cinnamon	9	2	310
Cinnamon	10	1	331
Cinnamon	10	2	295
Cumin	1	1	337
Cumin	1	2	214
Cumin	2	1	119
Cumin	2	2	514
Cumin	3	1	268
Cumin	3	2	364
Cumin	4	1	192
Cumin	4	2	343
Cumin	5	1	246
Cumin	5	2	308
Cumin	6	1	403
Cumin	6	2	337
Cumin	7	1	200
Cumin	7	2	190
Cumin	8	1	300
Cumin	8	2	438
Cumin	9	1	313
Cumin	9	2	229
Cumin	10	1	298
Cumin	10	2	331

Table B.2 Raw data for unirradiated test material – screening PSL using DOS system (thyme and alfalfa)

Thyme	1	1	376
Thyme	1	2	534
Thyme	2	1	409
Thyme	2	2	344
Thyme	3	1	342
Thyme	3	2	393
Thyme	4	1	524
Thyme	4	2	414
Thyme	5	1	468
Thyme	5	2	456
Thyme	6	1	499
Thyme	6	2	417
Thyme	7	1	385
Thyme	7	2	293
Thyme	8	1	447
Thyme	8	2	469
Thyme	9	1	391
Thyme	9	2	371
Thyme	10	1	356
Thyme	10	2	363
Alfalfa	1	1	1005
Alfalfa	1	2	1239
Alfalfa	2	1	1278
Alfalfa	2	2	2103
Alfalfa	3	1	1414
Alfalfa	3	2	1527
Alfalfa	4	1	1532
Alfalfa	4	2	1412
Alfalfa	5	1	1423
Alfalfa	5	2	1295
Alfalfa	6	1	1315
Alfalfa	6	2	1415
Alfalfa	7	1	1257
Alfalfa	7	2	1708
Alfalfa	8	1	1628
Alfalfa	8	2	1459
Alfalfa	9	1	1403
Alfalfa	9	2	1306
Alfalfa	10	1	1430
Alfalfa	10	2	918

Table B.3 Raw data for unirradiated test material – screening PSL using DOS system (ginseng and guarana)

Ginseng	1	1	50909
Ginseng	1	2	68823
Ginseng	2	1	43415
Ginseng	2	2	75078
Ginseng	3	1	51911
Ginseng	3	2	89701
Ginseng	4	1	47002
Ginseng	4	2	57697
Ginseng	5	1	68361
Ginseng	5	2	53836
Ginseng	6	1	96373
Ginseng	6	2	92770
Ginseng	7	1	130307
Ginseng	7	2	91508
Ginseng	8	1	104540
Ginseng	8	2	107935
Ginseng	9	1	75784
Ginseng	9	2	64673
Ginseng	10	1	85731
Ginseng	10	2	75324
Guarana	1	1	514
Guarana	1	2	405
Guarana	2	1	460
Guarana	2	2	404
Guarana	3	1	581
Guarana	3	2	420
Guarana	4	1	474
Guarana	4	2	418
Guarana	5	1	365
Guarana	5	2	537
Guarana	6	1	393
Guarana	6	2	515
Guarana	7	1	467
Guarana	7	2	351
Guarana	8	1	362
Guarana	8	2	308
Guarana	9	1	494
Guarana	9	2	1158
Guarana	10	1	482
Guarana	10	2	418

Table B.4 Raw data for unirradiated test material – screening PSL using Windows system (cinnamon and cumin)

Product	Pot	Aliquot	Terminal count
Cinnamon	1	3	474
Cinnamon	1	4	368
Cinnamon	2	3	433
Cinnamon	2	4	269
Cinnamon	3	3	468
Cinnamon	3	4	478
Cinnamon	4	3	368
Cinnamon	4	4	341
Cinnamon	5	3	353
Cinnamon	5	4	394
Cinnamon	6	3	231
Cinnamon	6	4	377
Cinnamon	7	3	330
Cinnamon	7	4	433
Cinnamon	8	3	358
Cinnamon	8	4	387
Cinnamon	9	3	285
Cinnamon	9	4	369
Cinnamon	10	3	311
Cinnamon	10	4	339
Cumin	1	3	355
Cumin	1	4	417
Cumin	2	3	366
Cumin	2	4	310
Cumin	3	3	160
Cumin	3	4	434
Cumin	4	3	383
Cumin	4	4	295
Cumin	5	3	408
Cumin	5	4	393
Cumin	6	3	393
Cumin	6	4	333
Cumin	7	3	363
Cumin	7	4	489
Cumin	8	3	347
Cumin	8	4	453
Cumin	9	3	473
Cumin	9	4	427
Cumin	10	3	260
Cumin	10	4	398

Table B.5 Raw data for unirradiated test material – screening PSL using Windows system (thyme and alfalfa)

Thyme	1	3	638
Thyme	1	4	636
Thyme	2	3	543
Thyme	2	4	545
Thyme	3	3	453
Thyme	3	4	597
Thyme	4	3	574
Thyme	4	4	546
Thyme	5	3	779
Thyme	5	4	578
Thyme	6	3	568
Thyme	6	4	585
Thyme	7	3	585
Thyme	7	4	532
Thyme	8	3	667
Thyme	8	4	623
Thyme	9	3	564
Thyme	9	4	640
Thyme	10	3	562
Thyme	10	4	445
Alfalfa	1	3	2410
Alfalfa	1	4	2231
Alfalfa	2	3	2444
Alfalfa	2	4	1483
Alfalfa	3	3	2191
Alfalfa	3	4	2016
Alfalfa	4	3	2044
Alfalfa	4	4	2497
Alfalfa	5	3	1952
Alfalfa	5	4	2992
Alfalfa	6	3	1858
Alfalfa	6	4	2522
Alfalfa	7	3	2130
Alfalfa	7	4	3224
Alfalfa	8	3	2246
Alfalfa	8	4	2034
Alfalfa	9	3	1901
Alfalfa	9	4	1882
Alfalfa	10	3	2233
Alfalfa	10	4	2074

Table B.6 Raw data for unirradiated test material – screening PSL using Windows system (ginseng and guarana)

Ginseng	1	3	129189
Ginseng	1	4	91097
Ginseng	2	3	115265
Ginseng	2	4	77381
Ginseng	3	3	107876
Ginseng	3	4	101006
Ginseng	4	3	98564
Ginseng	4	4	81304
Ginseng	5	3	84457
Ginseng	5	4	103023
Ginseng	6	3	136162
Ginseng	6	4	138554
Ginseng	7	3	218982
Ginseng	7	4	121338
Ginseng	8	3	139587
Ginseng	8	4	140621
Ginseng	9	3	112377
Ginseng	9	4	158222
Ginseng	10	3	109801
Ginseng	10	4	118690
Guarana	1	3	546
Guarana	1	4	631
Guarana	2	3	536
Guarana	2	4	524
Guarana	3	3	640
Guarana	3	4	588
Guarana	4	3	839
Guarana	4	4	839
Guarana	5	3	676
Guarana	5	4	518
Guarana	6	3	658
Guarana	6	4	576
Guarana	7	3	655
Guarana	7	4	563
Guarana	8	3	630
Guarana	8	4	564
Guarana	9	3	463
Guarana	9	4	606
Guarana	10	3	610
Guarana	10	4	594

Table B.7 Raw data for blended test material – screening PSL using DOS system (cinnamon and cumin)

	Product	Pot	Aliquot	Terminal count
10%	Cinnamon	1	1	247
	Cinnamon	1	2	343
	Cinnamon	2	1	354
	Cinnamon	2	2	339
	Cinnamon	3	1	507
	Cinnamon	3	2	368
	Cinnamon	4	1	266
	Cinnamon	4	2	244
	Cinnamon	5	1	357
	Cinnamon	5	2	621
	Cinnamon	6	1	498
	Cinnamon	6	2	717
	Cinnamon	7	1	339
	Cinnamon	7	2	346
	Cinnamon	8	1	328
	Cinnamon	8	2	349
	Cinnamon	9	1	688
	Cinnamon	9	2	283
	Cinnamon	10	1	403
	Cinnamon	10	2	333
1%	Cumin	1	1	3835
	Cumin	1	2	978
	Cumin	2	1	731
	Cumin	2	2	521
	Cumin	3	1	596
	Cumin	3	2	411
	Cumin	4	1	775
	Cumin	4	2	1046
	Cumin	5	1	477
	Cumin	5	2	591
	Cumin	6	1	740
	Cumin	6	2	800
	Cumin	7	1	1112
	Cumin	7	2	689
	Cumin	8	1	1666
	Cumin	8	2	436
	Cumin	9	1	825
	Cumin	9	2	603
	Cumin	10	1	552
	Cumin	10	2	598

Table B.8 Raw data for blended test material – screening PSL using DOS system (thyme and alfalfa)

0.10%	Thyme	1	1	749
	Thyme	1	2	1829
	Thyme	2	1	1271
	Thyme	2	2	1477
	Thyme	3	1	1064
	Thyme	3	2	427
	Thyme	4	1	1512
	Thyme	4	2	982
	Thyme	5	1	1834
	Thyme	5	2	406
	Thyme	6	1	677
	Thyme	6	2	1378
	Thyme	7	1	13650
	Thyme	7	2	816
	Thyme	8	1	1193
	Thyme	8	2	3576
	Thyme	9	1	654
	Thyme	9	2	3298
	Thyme	10	1	9098
	Thyme	10	2	3141
0.10%	Alfalfa	1	1	2525
	Alfalfa	1	2	2154
	Alfalfa	2	1	3063
	Alfalfa	2	2	2888
	Alfalfa	3	1	2075
	Alfalfa	3	2	1980
	Alfalfa	4	1	4318
	Alfalfa	4	2	3199
	Alfalfa	5	1	3005
	Alfalfa	5	2	3145
	Alfalfa	6	1	2434
	Alfalfa	6	2	7391
	Alfalfa	7	1	3464
	Alfalfa	7	2	2487
	Alfalfa	8	1	3769
	Alfalfa	8	2	3317
	Alfalfa	9	1	3384
	Alfalfa	9	2	2037
	Alfalfa	10	1	1631
	Alfalfa	10	2	3556

Table B.9 Raw data for blended test material – screening PSL using DOS system (green tea and guarana)

1%	Green Tea	1	1	915
	Green Tea	1	2	586
	Green Tea	2	1	493
	Green Tea	2	2	542
	Green Tea	3	1	460
	Green Tea	3	2	652
	Green Tea	4	1	733
	Green Tea	4	2	662
	Green Tea	5	1	563
	Green Tea	5	2	527
	Green Tea	6	1	430
	Green Tea	6	2	444
	Green Tea	7	1	489
	Green Tea	7	2	430
	Green Tea	8	1	998
	Green Tea	8	2	915
	Green Tea	9	1	613
	Green Tea	9	2	1161
	Green Tea	10	1	415
	Green Tea	10	2	613
10%	Guarana	1	1	971
	Guarana	1	2	1672
	Guarana	2	1	954
	Guarana	2	2	1343
	Guarana	3	1	2614
	Guarana	3	2	525
	Guarana	4	1	2875
	Guarana	4	2	835
	Guarana	5	1	1376
	Guarana	5	2	2508
	Guarana	6	1	1356
	Guarana	6	2	1118
	Guarana	7	1	1937
	Guarana	7	2	2394
	Guarana	8	1	1560
	Guarana	8	2	601
	Guarana	9	1	945
	Guarana	9	2	1613
	Guarana	10	1	2187
	Guarana	10	2	1502

Table B.10 Raw data for blended test material – screening PSL using Windows system (cinnamon and cumin)

	Product	Pot	Aliquot	Terminal count
10%	Cinnamon	1	3	469
	Cinnamon	1	4	438
	Cinnamon	2	3	380
	Cinnamon	2	4	392
	Cinnamon	3	3	428
	Cinnamon	3	4	577
	Cinnamon	4	3	308
	Cinnamon	4	4	598
	Cinnamon	5	3	452
	Cinnamon	5	4	404
	Cinnamon	6	3	417
	Cinnamon	6	4	559
	Cinnamon	7	3	393
	Cinnamon	7	4	639
	Cinnamon	8	3	722
	Cinnamon	8	4	461
	Cinnamon	9	3	499
	Cinnamon	9	4	619
	Cinnamon	10	3	492
	Cinnamon	10	4	453
1%	Cumin	1	3	1245
	Cumin	1	4	1249
	Cumin	2	3	1145
	Cumin	2	4	908
	Cumin	3	3	1497
	Cumin	3	4	1646
	Cumin	4	3	1185
	Cumin	4	4	840
	Cumin	5	3	3234
	Cumin	5	4	519
	Cumin	6	3	830
	Cumin	6	4	1284
	Cumin	7	3	1121
	Cumin	7	4	1724
	Cumin	8	3	10689
	Cumin	8	4	400389
	Cumin	9	3	1495
	Cumin	9	4	1472
	Cumin	10	3	931
	Cumin	10	4	1144

Table B.11 Raw data for blended test material – screening PSL using Windows system (thyme and alfalfa)

0.10%	Thyme	1	3	8056
	Thyme	1	4	682
	Thyme	2	3	556
	Thyme	2	4	2258
	Thyme	3	3	2511
	Thyme	3	4	13053
	Thyme	4	3	1477
	Thyme	4	4	10284
	Thyme	5	3	2219
	Thyme	5	4	3064
	Thyme	6	3	3401
	Thyme	6	4	8247
	Thyme	7	3	19851
	Thyme	7	4	687
	Thyme	8	3	1304
	Thyme	8	4	547
	Thyme	9	3	3769
	Thyme	9	4	1306
	Thyme	10	3	1756
	Thyme	10	4	906
0.10%	Alfalfa	1	3	5128
	Alfalfa	1	4	5171
	Alfalfa	2	3	4368
	Alfalfa	2	4	5471
	Alfalfa	3	3	4382
	Alfalfa	3	4	3932
	Alfalfa	4	3	3175
	Alfalfa	4	4	6657
	Alfalfa	5	3	3814
	Alfalfa	5	4	6882
	Alfalfa	6	3	4470
	Alfalfa	6	4	8809
	Alfalfa	7	3	5930
	Alfalfa	7	4	5528
	Alfalfa	8	3	3329
	Alfalfa	8	4	19123
	Alfalfa	9	3	2901
	Alfalfa	9	4	30426
	Alfalfa	10	3	5520
	Alfalfa	10	4	2993

Table B.12 Raw data for blended test material – screening PSL using Windows system (green tea and guarana)

1%	Green Tea	1	3	810
	Green Tea	1	4	1313
	Green Tea	2	3	663
	Green Tea	2	4	1627
	Green Tea	3	3	745
	Green Tea	3	4	742
	Green Tea	4	3	984
	Green Tea	4	4	695
	Green Tea	5	3	1113
	Green Tea	5	4	1105
	Green Tea	6	3	815
	Green Tea	6	4	669
	Green Tea	7	3	1300
	Green Tea	7	4	861
	Green Tea	8	3	613
	Green Tea	8	4	796
	Green Tea	9	3	4172
	Green Tea	9	4	654
	Green Tea	10	3	789
	Green Tea	10	4	1058
10%	Guarana	1	3	2214
	Guarana	1	4	1125
	Guarana	2	3	2155
	Guarana	2	4	1208
	Guarana	3	3	1239
	Guarana	3	4	986
	Guarana	4	3	876
	Guarana	4	4	25795
	Guarana	5	3	980
	Guarana	5	4	2702
	Guarana	6	3	969
	Guarana	6	4	2969
	Guarana	7	3	1659
	Guarana	7	4	3720
	Guarana	8	3	4157
	Guarana	8	4	1092
	Guarana	9	3	1731
	Guarana	9	4	1740
	Guarana	10	3	2560
	Guarana	10	4	2022

Table B.13 Raw data for irradiated test material – screening PSL using DOS system (cinnamon and cumin)

Product	Pot	Aliquot	Terminal count
Cinnamon	1	1	1011
Cinnamon	1	2	1256
Cinnamon	2	1	1012
Cinnamon	2	2	2001
Cinnamon	3	1	1170
Cinnamon	3	2	983
Cinnamon	4	1	846
Cinnamon	4	2	817
Cinnamon	5	1	785
Cinnamon	5	2	873
Cinnamon	6	1	750
Cinnamon	6	2	726
Cinnamon	7	1	1334
Cinnamon	7	2	1511
Cinnamon	8	1	684
Cinnamon	8	2	638
Cinnamon	9	1	986
Cinnamon	9	2	1079
Cinnamon	10	1	1222
Cinnamon	10	2	1217
Cumin	1	1	41295
Cumin	1	2	34538
Cumin	2	1	45305
Cumin	2	2	42922
Cumin	3	1	40221
Cumin	3	2	45723
Cumin	4	1	56727
Cumin	4	2	64987
Cumin	5	1	65728
Cumin	5	2	37595
Cumin	6	1	29165
Cumin	6	2	26293
Cumin	7	1	31399
Cumin	7	2	43050
Cumin	8	1	32253
Cumin	8	2	36765
Cumin	9	1	47837
Cumin	9	2	44950
Cumin	10	1	30438
Cumin	10	2	43247

Table B.14 Raw data for irradiated test material – screening PSL using DOS system (thyme and alfalfa)

Thyme	1	1	1247445
Thyme	1	2	1536936
Thyme	2	1	1310321
Thyme	2	2	1314929
Thyme	3	1	1299359
Thyme	3	2	1421613
Thyme	4	1	1359635
Thyme	4	2	1435998
Thyme	5	1	1431652
Thyme	5	2	1594659
Thyme	6	1	1084643
Thyme	6	2	1163323
Thyme	7	1	1421632
Thyme	7	2	1291258
Thyme	8	1	1163646
Thyme	8	2	1328137
Thyme	9	1	1445478
Thyme	9	2	1560624
Thyme	10	1	993451
Thyme	10	2	1667111
Alfalfa	1	1	1939199
Alfalfa	1	2	1696185
Alfalfa	2	1	1658370
Alfalfa	2	2	1885272
Alfalfa	3	1	1375169
Alfalfa	3	2	1637665
Alfalfa	4	1	1512037
Alfalfa	4	2	1401896
Alfalfa	5	1	1301580
Alfalfa	5	2	1849369
Alfalfa	6	1	1703206
Alfalfa	6	2	1762162
Alfalfa	7	1	1675984
Alfalfa	7	2	1544523
Alfalfa	8	1	1753102
Alfalfa	8	2	1833171
Alfalfa	9	1	1775626
Alfalfa	9	2	1691910
Alfalfa	10	1	1504670
Alfalfa	10	2	1819261

Table B.15 Raw data for irradiated test material – screening PSL using DOS system (ginseng and guarana)

Ginseng	1	1	1922133
Ginseng	1	2	1045412
Ginseng	2	1	1464388
Ginseng	2	2	1891815
Ginseng	3	1	1932277
Ginseng	3	2	2323355
Ginseng	4	1	914746
Ginseng	4	2	1363383
Ginseng	5	1	1606332
Ginseng	5	2	1829607
Ginseng	6	1	1435092
Ginseng	6	2	1407579
Ginseng	7	1	1929987
Ginseng	7	2	1863252
Ginseng	8	1	1998132
Ginseng	8	2	1483050
Ginseng	9	1	849214
Ginseng	9	2	1815097
Ginseng	10	1	1252366
Ginseng	10	2	1527934
Guarana	1	1	12139
Guarana	1	2	7773
Guarana	2	1	10874
Guarana	2	2	11668
Guarana	3	1	14336
Guarana	3	2	7461
Guarana	4	1	12005
Guarana	4	2	13459
Guarana	5	1	8897
Guarana	5	2	12699
Guarana	6	1	13128
Guarana	6	2	11479
Guarana	7	1	7673
Guarana	7	2	12105
Guarana	8	1	7439
Guarana	8	2	13649
Guarana	9	1	5444
Guarana	9	2	9111
Guarana	10	1	7587
Guarana	10	2	15882

Table B.16 Raw data for irradiated test material – screening PSL using Windows system (cinnamon and cumin)

Product	Pot	Aliquot	Terminal count
Cinnamon	1	3	1599
Cinnamon	1	4	2669
Cinnamon	2	3	1074
Cinnamon	2	4	1896
Cinnamon	3	3	1600
Cinnamon	3	4	1600
Cinnamon	4	3	953
Cinnamon	4	4	1367
Cinnamon	5	3	1190
Cinnamon	5	4	1894
Cinnamon	6	3	1032
Cinnamon	6	4	3966
Cinnamon	7	3	1914
Cinnamon	7	4	1444
Cinnamon	8	3	1490
Cinnamon	8	4	997
Cinnamon	9	3	1513
Cinnamon	9	4	1903
Cinnamon	10	3	1809
Cinnamon	10	4	2025
Cumin	1	3	7949
Cumin	1	4	15055
Cumin	2	3	77419
Cumin	2	4	83554
Cumin	3	3	73065
Cumin	3	4	66757
Cumin	4	3	55464
Cumin	4	4	118119
Cumin	5	3	54585
Cumin	5	4	70847
Cumin	6	3	55676
Cumin	6	4	45955
Cumin	7	3	56032
Cumin	7	4	78399
Cumin	8	3	75503
Cumin	8	4	93409
Cumin	9	3	58917
Cumin	9	4	56633
Cumin	10	3	206819
Cumin	10	4	63119

Table B.17 Raw data for irradiated test material – screening PSL using Windows system (thyme and alfalfa)

Thyme	1	3	2069455
Thyme	1	4	2069008
Thyme	2	3	2248284
Thyme	2	4	2471742
Thyme	3	3	2508346
Thyme	3	4	1530415
Thyme	4	3	2180624
Thyme	4	4	2536341
Thyme	5	3	2233566
Thyme	5	4	2586829
Thyme	6	3	1841603
Thyme	6	4	1936758
Thyme	7	3	2531174
Thyme	7	4	2263492
Thyme	8	3	1815384
Thyme	8	4	2318838
Thyme	9	3	2546432
Thyme	9	4	1842183
Thyme	10	3	1953127
Thyme	10	4	2013855
Alfalfa	1	3	3107322
Alfalfa	1	4	2770651
Alfalfa	2	3	2588987
Alfalfa	2	4	2824472
Alfalfa	3	3	1724994
Alfalfa	3	4	2784276
Alfalfa	4	3	2550665
Alfalfa	4	4	2157776
Alfalfa	5	3	1775753
Alfalfa	5	4	2030215
Alfalfa	6	3	1670752
Alfalfa	6	4	2658654
Alfalfa	7	3	2492277
Alfalfa	7	4	2297719
Alfalfa	8	3	2157336
Alfalfa	8	4	2490233
Alfalfa	9	3	2204604
Alfalfa	9	4	2814626
Alfalfa	10	3	3054677
Alfalfa	10	4	2691473

Table B.18 Raw data for irradiated test material – screening PSL using Windows system (ginseng and guarana)

Ginseng	1	3	3616323
Ginseng	1	4	3382685
Ginseng	2	3	1940457
Ginseng	2	4	3666509
Ginseng	3	3	3203901
Ginseng	3	4	2440864
Ginseng	4	3	1684067
Ginseng	4	4	3036002
Ginseng	5	3	3513277
Ginseng	5	4	1169437
Ginseng	6	3	2495140
Ginseng	6	4	2618863
Ginseng	7	3	1660155
Ginseng	7	4	3224782
Ginseng	8	3	1014894
Ginseng	8	4	1794268
Ginseng	9	3	2825996
Ginseng	9	4	3421778
Ginseng	10	3	2986238
Ginseng	10	4	2863761
Guarana	1	3	10432
Guarana	1	4	21336
Guarana	2	3	14822
Guarana	2	4	17045
Guarana	3	3	12450
Guarana	3	4	21585
Guarana	4	3	19678
Guarana	4	4	21943
Guarana	5	3	14772
Guarana	5	4	42131
Guarana	6	3	12725
Guarana	6	4	14951
Guarana	7	3	14043
Guarana	7	4	15823
Guarana	8	3	27499
Guarana	8	4	13805
Guarana	9	3	93113
Guarana	9	4	17622
Guarana	10	3	22154
Guarana	10	4	18146

Table B.19 Raw data for unirradiated test material – CalPSL using DOS system (cinnamon and cumin)

Product	Pot	Aliquot	Terminal count
Cinnamon	1	1	1013
Cinnamon	1	2	745
Cinnamon	2	1	555
Cinnamon	2	2	1049
Cinnamon	3	1	1287
Cinnamon	3	2	843
Cinnamon	4	1	703
Cinnamon	4	2	1147
Cinnamon	5	1	931
Cinnamon	5	2	729
Cinnamon	6	1	588
Cinnamon	6	2	966
Cinnamon	7	1	895
Cinnamon	7	2	1086
Cinnamon	8	1	831
Cinnamon	8	2	987
Cinnamon	9	1	633
Cinnamon	9	2	595
Cinnamon	10	1	670
Cinnamon	10	2	878
Cumin	1	1	20890
Cumin	1	2	25053
Cumin	2	1	15561
Cumin	2	2	19881
Cumin	3	1	25106
Cumin	3	2	13143
Cumin	4	1	20630
Cumin	4	2	19197
Cumin	5	1	18650
Cumin	5	2	16967
Cumin	6	1	22041
Cumin	6	2	20165
Cumin	7	1	21559
Cumin	7	2	16573
Cumin	8	1	19328
Cumin	8	2	33621
Cumin	9	1	25288
Cumin	9	2	16744
Cumin	10	1	24531
Cumin	10	2	25806

Table B.20 Raw data for unirradiated test material – CalPSL using DOS system (thyme and alfalfa)

Thyme	1	1	840732
Thyme	1	2	943817
Thyme	2	1	796338
Thyme	2	2	714778
Thyme	3	1	433854
Thyme	3	2	374715
Thyme	4	1	667443
Thyme	4	2	638031
Thyme	5	1	564181
Thyme	5	2	639140
Thyme	6	1	696112
Thyme	6	2	762988
Thyme	7	1	720472
Thyme	7	2	625892
Thyme	8	1	673171
Thyme	8	2	750191
Thyme	9	1	678508
Thyme	9	2	583776
Thyme	10	1	507284
Thyme	10	2	507179
Alfalfa	1	1	414776
Alfalfa	1	2	672314
Alfalfa	2	1	546856
Alfalfa	2	2	793329
Alfalfa	3	1	483843
Alfalfa	3	2	782563
Alfalfa	4	1	919583
Alfalfa	4	2	727898
Alfalfa	5	1	498136
Alfalfa	5	2	519370
Alfalfa	6	1	729692
Alfalfa	6	2	601906
Alfalfa	7	1	473207
Alfalfa	7	2	403307
Alfalfa	8	1	816380
Alfalfa	8	2	275843
Alfalfa	9	1	746009
Alfalfa	9	2	751621
Alfalfa	10	1	284299
Alfalfa	10	2	635516

Table B.21 Raw data for unirradiated test material – CalPSL using DOS system (ginseng and guarana)

Ginseng	1	1	771272
Ginseng	1	2	1137950
Ginseng	2	1	765483
Ginseng	2	2	1014204
Ginseng	3	1	741741
Ginseng	3	2	1078820
Ginseng	4	1	869713
Ginseng	4	2	877073
Ginseng	5	1	1022958
Ginseng	5	2	903371
Ginseng	6	1	986539
Ginseng	6	2	972450
Ginseng	7	1	10388546
Ginseng	7	2	829190
Ginseng	8	1	971016
Ginseng	8	2	1126995
Ginseng	9	1	770456
Ginseng	9	2	811309
Ginseng	10	1	933743
Ginseng	10	2	892616
Guarana	1	1	6475
Guarana	1	2	6186
Guarana	2	1	4643
Guarana	2	2	7271
Guarana	3	1	3986
Guarana	3	2	4627
Guarana	4	1	3175
Guarana	4	2	4730
Guarana	5	1	4669
Guarana	5	2	4520
Guarana	6	1	5524
Guarana	6	2	5850
Guarana	7	1	5279
Guarana	7	2	6955
Guarana	8	1	3542
Guarana	8	2	3467
Guarana	9	1	3008
Guarana	9	2	9084
Guarana	10	1	7451
Guarana	10	2	4884

Table B.22 Raw data for blended test material – CalPSL using DOS system (cinnamon and cumin)

Product	Pot	Aliquot	Terminal count
Cinnamon	1	1	1455
Cinnamon	1	2	804
Cinnamon	2	1	521
Cinnamon	2	2	833
Cinnamon	3	1	1337
Cinnamon	3	2	612
Cinnamon	4	1	568
Cinnamon	4	2	778
Cinnamon	5	1	622
Cinnamon	5	2	914
Cinnamon	6	1	738
Cinnamon	6	2	957
Cinnamon	7	1	668
Cinnamon	7	2	483
Cinnamon	8	1	991
Cinnamon	8	2	838
Cinnamon	9	1	640
Cinnamon	9	2	727
Cinnamon	10	1	598
Cinnamon	10	2	698
Cumin	1	1	30961
Cumin	1	2	26371
Cumin	2	1	19701
Cumin	2	2	18745
Cumin	3	1	19363
Cumin	3	2	19838
Cumin	4	1	23065
Cumin	4	2	18026
Cumin	5	1	18561
Cumin	5	2	14061
Cumin	6	1	28618
Cumin	6	2	22912
Cumin	7	1	18382
Cumin	7	2	15237
Cumin	8	1	23445
Cumin	8	2	14074
Cumin	9	1	20205
Cumin	9	2	16811
Cumin	10	1	21655
Cumin	10	2	23639

Table B.23 Raw data for blended test material – CalPSL using DOS system (thyme and alfalfa)

Thyme	1	1	398363
Thyme	1	2	371945
Thyme	2	1	430019
Thyme	2	2	693962
Thyme	3	1	434975
Thyme	3	2	389787
Thyme	4	1	650881
Thyme	4	2	368264
Thyme	5	1	560852
Thyme	5	2	561161
Thyme	6	1	590765
Thyme	6	2	725939
Thyme	7	1	496417
Thyme	7	2	520714
Thyme	8	1	620459
Thyme	8	2	580600
Thyme	9	1	506686
Thyme	9	2	523871
Thyme	10	1	588234
Thyme	10	2	522528
Alfalfa	1	1	585629
Alfalfa	1	2	272544
Alfalfa	2	1	865929
Alfalfa	2	2	385618
Alfalfa	3	1	561471
Alfalfa	3	2	354716
Alfalfa	4	1	554405
Alfalfa	4	2	411671
Alfalfa	5	1	637168
Alfalfa	5	2	407438
Alfalfa	6	1	493682
Alfalfa	6	2	690026
Alfalfa	7	1	667635
Alfalfa	7	2	474318
Alfalfa	8	1	698663
Alfalfa	8	2	436820
Alfalfa	9	1	396748
Alfalfa	9	2	399833
Alfalfa	10	1	519481
Alfalfa	10	2	500224

Table B.24 Raw data for blended test material – CalPSL using DOS system (green tea and guarana)

Green Tea	1	1	15963
Green Tea	1	2	18127
Green Tea	2	1	16457
Green Tea	2	2	20707
Green Tea	3	1	15480
Green Tea	3	2	17624
Green Tea	4	1	14688
Green Tea	4	2	18319
Green Tea	5	1	17227
Green Tea	5	2	18078
Green Tea	6	1	13966
Green Tea	6	2	15293
Green Tea	7	1	13671
Green Tea	7	2	22493
Green Tea	8	1	13832
Green Tea	8	2	19335
Green Tea	9	1	14493
Green Tea	9	2	11865
Green Tea	10	1	8452
Green Tea	10	2	11747
Guarana	1	1	4851
Guarana	1	2	6553
Guarana	2	1	4734
Guarana	2	2	3748
Guarana	3	1	6220
Guarana	3	2	3146
Guarana	4	1	3832
Guarana	4	2	4074
Guarana	5	1	4916
Guarana	5	2	5225
Guarana	6	1	3818
Guarana	6	2	5542
Guarana	7	1	4117
Guarana	7	2	3708
Guarana	8	1	5831
Guarana	8	2	3128
Guarana	9	1	4865
Guarana	9	2	3684
Guarana	10	1	3527
Guarana	10	2	3546

Table B.25 Raw data for irradiated test material – CalPSL using DOS system (cinnamon and cumin)

Cinnamon	1	1	868
Cinnamon	1	2	1990
Cinnamon	2	1	1005
Cinnamon	2	2	2035
Cinnamon	3	1	1061
Cinnamon	3	2	1153
Cinnamon	4	1	1029
Cinnamon	4	2	1036
Cinnamon	5	1	881
Cinnamon	5	2	1177
Cinnamon	6	1	762
Cinnamon	6	2	1251
Cinnamon	7	1	1564
Cinnamon	7	2	2022
Cinnamon	8	1	714
Cinnamon	8	2	773
Cinnamon	9	1	1200
Cinnamon	9	2	866
Cinnamon	10	1	1687
Cinnamon	10	2	1053
Cumin	1	1	30588
Cumin	1	2	26578
Cumin	2	1	42707
Cumin	2	2	52546
Cumin	3	1	31844
Cumin	3	2	37487
Cumin	4	1	33718
Cumin	4	2	49699
Cumin	5	1	71516
Cumin	5	2	37973
Cumin	6	1	32379
Cumin	6	2	30949
Cumin	7	1	35912
Cumin	7	2	43786
Cumin	8	1	34134
Cumin	8	2	37394
Cumin	9	1	59393
Cumin	9	2	32960
Cumin	10	1	28320
Cumin	10	2	46835

Table B.26 Raw data for irradiated test material – CalPSL using DOS system (thyme and alfalfa)

Thyme	1	1	1012671
Thyme	1	2	1209843
Thyme	2	1	1190460
Thyme	2	2	1143372
Thyme	3	1	1254562
Thyme	3	2	1216127
Thyme	4	1	1251319
Thyme	4	2	1228436
Thyme	5	1	1212562
Thyme	5	2	1373835
Thyme	6	1	926664
Thyme	6	2	810714
Thyme	7	1	1256756
Thyme	7	2	1083122
Thyme	8	1	1039412
Thyme	8	2	1157156
Thyme	9	1	1256868
Thyme	9	2	1307798
Thyme	10	1	855692
Thyme	10	2	1365599
Alfalfa	1	1	1397299
Alfalfa	1	2	1373226
Alfalfa	2	1	1145463
Alfalfa	2	2	1341158
Alfalfa	3	1	1046030
Alfalfa	3	2	1202157
Alfalfa	4	1	1155443
Alfalfa	4	2	980252
Alfalfa	5	1	896726
Alfalfa	5	2	1324229
Alfalfa	6	1	1228162
Alfalfa	6	2	1315349
Alfalfa	7	1	1099776
Alfalfa	7	2	1072747
Alfalfa	8	1	1194021
Alfalfa	8	2	1389355
Alfalfa	9	1	1186718
Alfalfa	9	2	1181366
Alfalfa	10	1	993614
Alfalfa	10	2	1243627

Table B.27 Raw data for irradiated test material – CalPSL using DOS system (ginseng and guarana)

Ginseng	1	1	1618085
Ginseng	1	2	994530
Ginseng	2	1	1405917
Ginseng	2	2	1881509
Ginseng	3	1	1881444
Ginseng	3	2	1980817
Ginseng	4	1	811205
Ginseng	4	2	1109560
Ginseng	5	1	1389956
Ginseng	5	2	1447716
Ginseng	6	1	1208988
Ginseng	6	2	1178637
Ginseng	7	1	1762998
Ginseng	7	2	1599408
Ginseng	8	1	1829852
Ginseng	8	2	1181705
Ginseng	9	1	777623
Ginseng	9	2	1559750
Ginseng	10	1	1185151
Ginseng	10	2	1296406
Guarana	1	1	12697
Guarana	1	2	8803
Guarana	2	1	14450
Guarana	2	2	10317
Guarana	3	1	15984
Guarana	3	2	7607
Guarana	4	1	8844
Guarana	4	2	11289
Guarana	5	1	6471
Guarana	5	2	8074
Guarana	6	1	11993
Guarana	6	2	11180
Guarana	7	1	6695
Guarana	7	2	17801
Guarana	8	1	6963
Guarana	8	2	8956
Guarana	9	1	7771
Guarana	9	2	10301
Guarana	10	1	12242
Guarana	10	2	9958

Table B.28 Reference values for TL Homogeneity Testing

Product	Status	Mean G1/G2	SD G1/G2	CV G1/2
Cinnamon	U	0.021	0.025	121.02%
Cumin	U	0.029	0.038	132.83%
Thyme	U	0.002	0.001	53.26%
Ginseng	U	0.121	0.035	29.01%
Alfalfa	U	0.003	0.002	54.77%
Guarana	U	0.021	0.021	100.50%
Cinnamon	B	0.558	0.829	148.73%
Cumin	B	0.093	0.179	191.89%
Thyme	B	0.009	0.004	40.70%
Alfalfa	B	0.021	0.061	296.13%
Green Tea	B	0.062	0.147	238.67%
Guarana	B	0.583	0.237	40.58%
Cinnamon	I	2.145	0.988	46.07%
Cumin	I	2.646	0.865	32.68%
Thyme	I	2.494	0.369	14.78%
Ginseng	I	2.459	0.349	14.18%
Alfalfa	I	2.178	0.232	10.67%
Guarana	I	5.531	1.919	34.70%

APPENDIX C - PARTICIPANTS' RAW DATA

			Lab 1		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	290	377	negative
SP10897	Cumin	U	404	388	negative
SP10902	Thyme	U	519	601	negative
SP10950	Ginseng	U	71296	56612	positive
SP10951	Alfalfa	U	1590	1440	intermediate
SP10954	Guarana	U	1060	582	negative
SP10895	Cinnamon	I	1400	3593	intermediate
SP10897	Cumin	I	56094	49936	positive
SP10902	Thyme	I	2300406	2083482	positive
SP10950	Ginseng	I	1788648	2015894	positive
SP10951	Alfalfa	I	1627443	1672996	positive
SP10954	Guarana	I	8653	22095	positive
SP10895	Cinnamon	B	382	426	negative
SP10897	Cumin	B	656	423	negative
SP10902	Thyme	B	6864	5138	positive
SP10951	Alfalfa	B	3870	2226	intermediate
SP10952	Green tea	B	695	1244	negative
SP10954	Guarana	B	1353	1476	intermediate

			Lab2		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	243	250	negative
SP10897	Cumin	U	379	357	negative
SP10902	Thyme	U	577	239	negative
SP10950	Ginseng	U	43675	48600	positive
SP10951	Alfalfa	U	1417	788	intermediate
SP10954	Guarana	U	443	579	negative
SP10895	Cinnamon	I	1510	2035	intermediate
SP10897	Cumin	I	51243	58056	positive
SP10902	Thyme	I	2387916	2244157	positive
SP10950	Ginseng	I	1069455	895731	positive
SP10951	Alfalfa	I	991084	1058530	positive
SP10954	Guarana	I	9162	15912	positive
SP10895	Cinnamon	B	417	226	negative
SP10897	Cumin	B	847	861	intermediate
SP10902	Thyme	B	2443	3956	intermediate
SP10951	Alfalfa	B	3295	1791	intermediate
SP10952	Green tea	B	599	629	negative
SP10954	Guarana	B	2585	1613	intermediate

Table C.1 PSL screening data for lab 1 and lab 2

			Lab 3		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	317	298	negative
SP10897	Cumin	U	549	350	negative
SP10902	Thyme	U	369	540	negative
SP10950	Ginseng	U	64610	57907	positive
SP10951	Alfalfa	U	1064	1034	intermediate
SP10954	Guarana	U	551	395	negative
SP10895	Cinnamon	I	1294	1118	intermediate
SP10897	Cumin	I	32163	36021	positive
SP10902	Thyme	I	1860403	1687860	positive
SP10950	Ginseng	I	877302	896820	positive
SP10951	Alfalfa	I	1189904	893193	positive
SP10954	Guarana	I	9501	13055	positive
SP10895	Cinnamon	B	438	350	negative
SP10897	Cumin	B	937	721	intermediate
SP10902	Thyme	B	2886	3018	intermediate
SP10951	Alfalfa	B	3035	2480	intermediate
SP10952	Green tea	B	970	475	intermediate
SP10954	Guarana	B	1059	994	intermediate

			Lab 4		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	292	301	negative
SP10897	Cumin	U	230	249	negative
SP10902	Thyme	U	356	494	negative
SP10950	Ginseng	U	26682	20681	positive
SP10951	Alfalfa	U	1031	1134	intermediate
SP10954	Guarana	U	394	417	negative
SP10895	Cinnamon	I	1445	1065	intermediate
SP10897	Cumin	I	36529	23974	positive
SP10902	Thyme	I	1525609	1055437	positive
SP10950	Ginseng	I	1261185	866445	positive
SP10951	Alfalfa	I	946336	602226	positive
SP10954	Guarana	I	6000	4578	positive
SP10895	Cinnamon	B	378	419	negative
SP10897	Cumin	B	589	547	negative
SP10902	Thyme	B	615	623	negative
SP10951	Alfalfa	B	3976	2747	intermediate
SP10952	Green tea	B	845	656	intermediate
SP10954	Guarana	B	1831	1448	intermediate

Table C.2 PSL screening data for lab 3 and lab 4

			Lab 5	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	115	522
SP10897	Cumin	U	252	357
SP10902	Thyme	U	456	378
SP10950	Ginseng	U	63417	49668
SP10951	Alfalfa	U	1154	1359
SP10954	Guarana	U	800	532
SP10895	Cinnamon	I	1596	2023
SP10897	Cumin	I	66135	57943
SP10902	Thyme	I	2219630	2081064
SP10950	Ginseng	I	1773460	1802351
SP10951	Alfalfa	I	1496767	1641179
SP10954	Guarana	I	26202	14213
SP10895	Cinnamon	B	380	365
SP10897	Cumin	B	559	558
SP10902	Thyme	B	1111	5434
SP10951	Alfalfa	B	4782	4045
SP10952	Green tea	B	637	623
SP10954	Guarana	B	1041	1609

			Lab 6	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	348	321
SP10897	Cumin	U	478	563
SP10902	Thyme	U	440	680
SP10950	Ginseng	U	77175	61513
SP10951	Alfalfa	U	2097	2087
SP10954	Guarana	U	464	1075
SP10895	Cinnamon	I	1982	1934
SP10897	Cumin	I	79153	48082
SP10902	Thyme	I	2496495	2617106
SP10950	Ginseng	I	2148308	1464602
SP10951	Alfalfa	I	1567742	1339934
SP10954	Guarana	I	12730	20230
SP10895	Cinnamon	B	466	339
SP10897	Cumin	B	1606	1368
SP10902	Thyme	B	1766	808
SP10951	Alfalfa	B	4926	3558
SP10952	Green tea	B	446	2790
SP10954	Guarana	B	1116	1053

Table C.3 PSL screening data for lab 5 and lab 6

			Lab 7		
Sample	Description	Status	Pot 1	Pot 2	Evaluations
SP10895	Cinnamon	U	314	366	no evidence of irradiation
SP10897	Cumin	U	322	391	no evidence of irradiation
SP10902	Thyme	U	727	584	possibly irradiated
SP10950	Ginseng	U	64482	78342	likely to be irradiated
SP10951	Alfalfa	U	2079	1630	possibly irradiated
SP10954	Guarana	U	369	786	possibly irradiated
SP10895	Cinnamon	I	1806	1458	possibly irradiated
SP10897	Cumin	I	70724	54039	likely to be irradiated
SP10902	Thyme	I	2839541	2871106	likely to be irradiated
SP10950	Ginseng	I	1566045	2036049	likely to be irradiated
SP10951	Alfalfa	I	1580077	1494325	likely to be irradiated
SP10954	Guarana	I	26757	12051	likely to be irradiated
SP10895	Cinnamon	B	639	316	no evidence of irradiation
SP10897	Cumin	B	1029	770	possibly irradiated
SP10902	Thyme	B	7600	7534	likely to be irradiated
SP10951	Alfalfa	B	3409	5298	likely to be irradiated
SP10952	Green tea	B	986	884	possibly irradiated
SP10954	Guarana	B	3424	1489	possibly irradiated

Calibrated

			Lab 7	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	2093	2959
SP10897	Cumin	U	38336	43511
SP10902	Thyme	U	1308837	1255158
SP10950	Ginseng	U	643026	914666
SP10951	Alfalfa	U	613969	626091
SP10954	Guarana	U	5466	10223
SP10895	Cinnamon	I	4065	4701
SP10897	Cumin	I	51012	43180
SP10902	Thyme	I	2089473	2098645
SP10950	Ginseng	I	1109859	1454262
SP10951	Alfalfa	I	1025726	938468
SP10954	Guarana	I	21121	10841
SP10895	Cinnamon	B	2148	4040
SP10897	Cumin	B	35563	35168
SP10902	Thyme	B	1401938	1367553
SP10951	Alfalfa	B	867098	1127647
SP10952	Green tea	B	21711	20413
SP10954	Guarana	B	11963	11184

Table C.4 PSL screening and CalPSL data for lab 7

			Lab 8		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	248	349	negative
SP10897	Cumin	U	310	258	negative
SP10902	Thyme	U	488	553	negative
SP10950	Ginseng	U	82170	84426	positive
SP10951	Alfalfa	U	1823	1318	intermediate
SP10954	Guarana	U	664	849	intermediate*
SP10895	Cinnamon	I	1531	2775	intermediate
SP10897	Cumin	I	54046	64707	positive
SP10902	Thyme	I	2330655	2249628	positive
SP10950	Ginseng	I	1377069	1573168	positive
SP10951	Alfalfa	I	1731173	1470757	positive
SP10954	Guarana	I	10844	11514	positive
SP10895	Cinnamon	B	395	443	negative
SP10897	Cumin	B	785	1389	intermediate
SP10902	Thyme	B	12927	4259	positive*
SP10951	Alfalfa	B	2969	7061	positive*
SP10952	Green tea	B	654	1887	intermediate*
SP10954	Guarana	B	1068	2892	intermediate

* based on higher aliquot

			Lab 9	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	333	424
SP10897	Cumin	U	445	413
SP10902	Thyme	U	503	526
SP10950	Ginseng	U	66763	69292
SP10951	Alfalfa	U	1554	4396
SP10954	Guarana	U	463	370
SP10895	Cinnamon	I	2725	1956
SP10897	Cumin	I	63419	50573
SP10902	Thyme	I	2359159	2434121
SP10950	Ginseng	I	1889087	1495724
SP10951	Alfalfa	I	1633497	1847160
SP10954	Guarana	I	11650	10770
SP10895	Cinnamon	B	686	515
SP10897	Cumin	B	1179	738
SP10902	Thyme	B	5382	10722
SP10951	Alfalfa	B	445	8386
SP10952	Green tea	B	744	530
SP10954	Guarana	B	3821	2359

Table C.5 PSL screening data for lab 8 and lab 9

			Lab 10	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	387	620
SP10897	Cumin	U	1163	967
SP10902	Thyme	U	645	690
SP10950	Ginseng	U	93821	107082
SP10951	Alfalfa	U	2026	1454
SP10954	Guarana	U	625	537
SP10895	Cinnamon	I	2804	2054
SP10897	Cumin	I	90675	56533
SP10902	Thyme	I	3257455	3291994
SP10950	Ginseng	I	2545502	2382230
SP10951	Alfalfa	I	2195264	1863379
SP10954	Guarana	I	11822	26359
SP10895	Cinnamon	B	823	734
SP10897	Cumin	B	1438	1124
SP10902	Thyme	B	8734	1248
SP10951	Alfalfa	B	9170	4206
SP10952	Green tea	B	811	1210
SP10954	Guarana	B	1132	1947

			Lab 11		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	418	335	no evidence of treatment
SP10897	Cumin	U	521	264	no evidence of treatment
SP10902	Thyme	U	644	463	no evidence of treatment
SP10950	Ginseng	U	75986	63325	likely to be irradiated
SP10951	Alfalfa	U	2133	1407	possibly irradiated
SP10954	Guarana	U	537	629	no evidence of treatment
SP10895	Cinnamon	I	1181	1641	possibly irradiated
SP10897	Cumin	I	49788	45959	likely to be irradiated
SP10902	Thyme	I	2048890	2180616	likely to be irradiated
SP10950	Ginseng	I	1225741	1275578	likely to be irradiated
SP10951	Alfalfa	I	1499096	1400154	likely to be irradiated
SP10954	Guarana	I	10132	7764	likely to be irradiated
SP10895	Cinnamon	B	414	331	no evidence of treatment
SP10897	Cumin	B	677	633	no evidence of treatment
SP10902	Thyme	B	1044	5465	possibly irradiated
SP10951	Alfalfa	B	3225	3712	possibly irradiated
SP10952	Green tea	B	403	839	possibly irradiated
SP10954	Guarana	B	1311	1411	possibly irradiated

Table C.6 PSL screening data for lab 10 and lab 11

			Lab 13	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	8003	7976
SP10897	Cumin	U	451	278
SP10902	Thyme	U	2753	2004
SP10950	Ginseng	U	75037	72652
SP10951	Alfalfa	U	2102	2793
SP10954	Guarana	U	4723	4682
SP10895	Cinnamon	I	2514	1682
SP10897	Cumin	I	75688	61489
SP10902	Thyme	I	2756530	2825349
SP10950	Ginseng	I	2053791	2494282
SP10951	Alfalfa	I	2167841	2471180
SP10954	Guarana	I	18296	14457
SP10895	Cinnamon	B	645	512
SP10897	Cumin	B	1725	1573
SP10902	Thyme	B	1814	1505
SP10951	Alfalfa	B	3755	4900
SP10952	Green tea	B	770	696
SP10954	Guarana	B	20712	15822

			Lab 14	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	309	343
SP10897	Cumin	U	725	690
SP10902	Thyme	U	509	429
SP10950	Ginseng	U	30185	32851
SP10951	Alfalfa	U	1189	1076
SP10954	Guarana	U	517	548
SP10895	Cinnamon	I	1723	1322
SP10897	Cumin	I	43675	37685
SP10902	Thyme	I	1886012	1629896
SP10950	Ginseng	I	1099052	937135
SP10951	Alfalfa	I	1029908	934779
SP10954	Guarana	I	13726	8230
SP10895	Cinnamon	B	511	504
SP10897	Cumin	B	750	807
SP10902	Thyme	B	6361	4378
SP10951	Alfalfa	B	3718	2378
SP10952	Green tea	B	537	492
SP10954	Guarana	B	1490	1209

Table C.7 PSL screening data for lab 13 and lab 14

			Lab 15	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	383	319
SP10897	Cumin	U	312	473
SP10902	Thyme	U	693	486
SP10950	Ginseng	U	24521	20897
SP10951	Alfalfa	U	1097	837
SP10954	Guarana	U	351	576
SP10895	Cinnamon	I	930	1068
SP10897	Cumin	I	17399	24022
SP10902	Thyme	I	1492625	1554656
SP10950	Ginseng	I	840978	729728
SP10951	Alfalfa	I	845658	838252
SP10954	Guarana	I	4711	6860
SP10895	Cinnamon	B	302	307
SP10897	Cumin	B	397	568
SP10902	Thyme	B	1590	2651
SP10951	Alfalfa	B	1433	1820
SP10952	Green tea	B	536	548
SP10954	Guarana	B	1565	1115

Table C.8 PSL screening data for lab 15

			Lab 16		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	327	330	negative
SP10897	Cumin	U	573	335	negative
SP10902	Thyme	U	535	529	negative
SP10950	Ginseng	U	68443	71745	positive
SP10951	Alfalfa	U	1461	1759	intermediate
SP10954	Guarana	U	388	674	negative
SP10895	Cinnamon	I	1434	1782	intermediate
SP10897	Cumin	I	97680	53998	positive
SP10902	Thyme	I	2445169	2297228	positive
SP10950	Ginseng	I	1236713	1200428	positive
SP10951	Alfalfa	I	1588804	1315758	positive
SP10954	Guarana	I	8748	14589	positive
SP10895	Cinnamon	B	468	596	negative
SP10897	Cumin	B	1207	3686	intermediate
SP10902	Thyme	B	5765	1033	positive
SP10951	Alfalfa	B	5972	4522	positive
SP10952	Green tea	B	906	520	intermediate
SP10954	Guarana	B	1227	1676	intermediate

Calibrated

			Lab16	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	891	976
SP10897	Cumin	U	26681	91461
SP10902	Thyme	U	1098699	1156171
SP10950	Ginseng	U	597253	597717
SP10951	Alfalfa	U	794660	690255
SP10954	Guarana	U	7895	55724
SP10895	Cinnamon	I	1637	1115
SP10897	Cumin	I	67592	41347
SP10902	Thyme	I	1919561	1904405
SP10950	Ginseng	I	882547	912882
SP10951	Alfalfa	I	1067871	913664
SP10954	Guarana	I	7132	11883
SP10895	Cinnamon	B	1046	1540
SP10897	Cumin	B	26352	32379
SP10902	Thyme	B	1087418	1079464
SP10951	Alfalfa	B	872357	734286
SP10952	Green tea	B	65974	26474
SP10954	Guarana	B	4702	6077

Table C.9 PSL screening and CalPSL data for lab 16

			Lab 17		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	306	352	negative
SP10897	Cumin	U	390	323	negative
SP10902	Thyme	U	540	554	negative
SP10950	Ginseng	U	74204	70320	positive
SP10951	Alfalfa	U	1153	1682	intermediate
SP10954	Guarana	U	658	491	negative
SP10895	Cinnamon	I	1764	1472	intermediate
SP10897	Cumin	I	61157	52292	positive
SP10902	Thyme	I	2413105	2529445	positive
SP10950	Ginseng	I	1779836	1880891	positive
SP10951	Alfalfa	I	2052144	1959145	positive
SP10954	Guarana	I	58198	69171	positive
SP10895	Cinnamon	B	558	446	negative
SP10897	Cumin	B	994	1243	intermediate
SP10902	Thyme	B	7931	13825	positive
SP10951	Alfalfa	B	7954	6942	positive
SP10952	Green tea	B	505	450	negative
SP10954	Guarana	B	1128	1783	intermediate

			Lab 18	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	400	225
SP10897	Cumin	U	496	259
SP10902	Thyme	U	354	433
SP10950	Ginseng	U	25793	17956
SP10951	Alfalfa	U	772	709
SP10954	Guarana	U	433	434
SP10895	Cinnamon	I	774	1059
SP10897	Cumin	I	22114	19108
SP10902	Thyme	I	832739	818471
SP10950	Ginseng	I	461142	343413
SP10951	Alfalfa	I	373889	442886
SP10954	Guarana	I	2646	3840
SP10895	Cinnamon	B	369	292
SP10897	Cumin	B	423	370
SP10902	Thyme	B	545	1008
SP10951	Alfalfa	B	1477	1779
SP10952	Green tea	B	369	499
SP10954	Guarana	B	368	550

Table C.10 PSL screening data for lab 17 and lab 18

			Lab 19		
Sample	Description	Status	Pot 1	Pot 2	Evaulation
SP10895	Cinnamon	U	400	369	negative
SP10897	Cumin	U	228	298	negative
SP10902	Thyme	U	414	479	negative
SP10950	Ginseng	U	46839	57990	positive
SP10951	Alfalfa	U	1463	1640	intermediate
SP10954	Guarana	U	446	601	negative
SP10895	Cinnamon	I	1407	2660	intermediate
SP10897	Cumin	I	48031	63182	positive
SP10902	Thyme	I	2102056	2098267	positive
SP10950	Ginseng	I	1496446	1612183	positive
SP10951	Alfalfa	I	1406762	1363460	positive
SP10954	Guarana	I	16379	8819	positive
SP10895	Cinnamon	B	413	271	negative
SP10897	Cumin	B	1313	648	intermediate
SP10902	Thyme	B	737	911	intermediate
SP10951	Alfalfa	B	4512	3118	intermediate
SP10952	Green tea	B	547	585	negative
SP10954	Guarana	B	2630	1042	intermediate

			Lab 20		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	363	439	unirradiated
SP10897	Cumin	U	162	296	unirradiated
SP10902	Thyme	U	451	616	unirradiated
SP10950	Ginseng	U	112669	115693	indication of irradiation
SP10951	Alfalfa	U	1758	2592	determination not possible
SP10954	Guarana	U	906	1158	determination not possible
SP10895	Cinnamon	I	1470	1540	determination not possible
SP10897	Cumin	I	73003	64185	indication of irradiation
SP10902	Thyme	I	2807962	3098111	indication of irradiation
SP10950	Ginseng	I	2399546	2133380	indication of irradiation
SP10951	Alfalfa	I	2918376	2105431	indication of irradiation
SP10954	Guarana	I	35077	17071	indication of irradiation
SP10895	Cinnamon	B	373	293	unirradiated
SP10897	Cumin	B	754	1035	determination not possible
SP10902	Thyme	B	9312	1466	determination not possible
SP10951	Alfalfa	B	4187	2842	determination not possible
SP10952	Green tea	B	974	725	determination not possible
SP10954	Guarana	B	3421	2355	determination not possible

Table C.11 PSL screening data for lab 19 and lab 20

			Lab 21		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	361	380	untreated
SP10897	Cumin	U	489	867	untreated
SP10902	Thyme	U	590	572	untreated
SP10950	Ginseng	U	68554	70526	Irradiated
SP10951	Alfalfa	U	1330	1173	mixture
SP10954	Guarana	U	551	547	untreated
SP10895	Cinnamon	I	1012	2334	mixture
SP10897	Cumin	I	48100	38382	Irradiated
SP10902	Thyme	I	2039543	1877678	Irradiated
SP10950	Ginseng	I	1448532	1534095	Irradiated
SP10951	Alfalfa	I	1088751	949471	Irradiated
SP10954	Guarana	I	8805	10871	Irradiated
SP10895	Cinnamon	B	561	382	untreated
SP10897	Cumin	B	1203	552	mixture
SP10902	Thyme	B	8663	1701	mixture
SP10951	Alfalfa	B	1940	5680	Irradiated
SP10952	Green tea	B	1114	630	untreated
SP10954	Guarana	B	1530	1229	mixture

Calibrated

			Lab 21	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	4420	3085
SP10897	Cumin	U	109707	27058
SP10902	Thyme	U	928854	912027
SP10950	Ginseng	U	696899	786331
SP10951	Alfalfa	U	506260	395009
SP10954	Guarana	U	10069	6558
SP10895	Cinnamon	I	4404	3564
SP10897	Cumin	I	36705	31818
SP10902	Thyme	I	1585612	1434291
SP10950	Ginseng	I	1025109	1093708
SP10951	Alfalfa	I	1153842	1048614
SP10954	Guarana	I	13519	11124
SP10895	Cinnamon	B	3366	5348
SP10897	Cumin	B	35009	21413
SP10902	Thyme	B	888656	988969
SP10951	Alfalfa	B	608640	594829
SP10952	Green tea	B	12128	14695
SP10954	Guarana	B	10748	7830

Table C.12 PSL screening and CalPSL data for lab 21

			Lab 22		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	274	332	negative
SP10897	Cumin	U	222	464	negative
SP10902	Thyme	U	513	423	negative
SP10950	Ginseng	U	47068	47706	positive
SP10951	Alfalfa	U	1172	985	intermediate
SP10954	Guarana	U	418	671	negative
SP10895	Cinnamon	I	1079	1164	intermediate
SP10897	Cumin	I	45945	30929	positive
SP10902	Thyme	I	1696786	1564969	positive
SP10950	Ginseng	I	927967	1195451	positive
SP10951	Alfalfa	I	1376963	1241872	positive
SP10954	Guarana	I	9882	7756	positive
SP10895	Cinnamon	B	261	537	negative
SP10897	Cumin	B	570	720	intermediate
SP10902	Thyme	B	641	1338	intermediate
SP10951	Alfalfa	B	2277	5702	intermediate
SP10952	Green tea	B	386	506	negative
SP10954	Guarana	B	740	1326	intermediate

Table C.13 PSL screening data for lab 22

			Lab 23	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	214	338
SP10897	Cumin	U	459	288
SP10902	Thyme	U	300	448
SP10950	Ginseng	U	63085	44861
SP10951	Alfalfa	U	1142	1277
SP10954	Guarana	U	557	654
SP10895	Cinnamon	I	1309	649
SP10897	Cumin	I	36623	34316
SP10902	Thyme	I	1487175	1539358
SP10950	Ginseng	I	810093	1154584
SP10951	Alfalfa	I	924463	917881
SP10954	Guarana	I	5916	9377
SP10895	Cinnamon	B	356	421
SP10897	Cumin	B	457	101
SP10902	Thyme	B	1426	578
SP10951	Alfalfa	B	1674	2222
SP10952	Green tea	B	464	710
SP10954	Guarana	B	1456	1336

Calibrated

			Lab 23		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	564	315	unclear (non detectable)
SP10897	Cumin	U	5068	5623	non irradiated
SP10902	Thyme	U	302214	320209	non irradiated
SP10950	Ginseng	U	166511	198948	irradiated
SP10951	Alfalfa	U	147471	193566	might contain irradiated components
SP10954	Guarana	U	2520	2954	non irradiated
SP10895	Cinnamon	I	682	539	might contain irradiated components
SP10897	Cumin	I	7388	8019	irradiated
SP10902	Thyme	I	438152	355601	irradiated
SP10950	Ginseng	I	223164	389885	irradiated
SP10951	Alfalfa	I	267497	216312	irradiated
SP10954	Guarana	I	2313	3103	irradiated
SP10895	Cinnamon	B	206	648	unclear (non detectable)
SP10897	Cumin	B	5489	5619	non irradiated
SP10902	Thyme	B	244579	273045	non irradiated
SP10951	Alfalfa	B	217001	156831	might contain irradiated components
SP10952	Green tea	B	10736	5463	non irradiated
SP10954	Guarana	B	4731	4855	might contain irradiated components

Table C.14 PSL screening and CalPSL data for lab 23

			Lab 24		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	296	329	not irradiated
SP10897	Cumin	U	262	200	not irradiated
SP10902	Thyme	U	361	362	not irradiated
SP10950	Ginseng	U	61760	81338	irradiated
SP10951	Alfalfa	U	1805	1753	
SP10954	Guarana	U	452	218	not irradiated
SP10895	Cinnamon	I	915	904	
SP10897	Cumin	I	50171	42222	irradiated
SP10902	Thyme	I	1458711	1851472	irradiated
SP10950	Ginseng	I	1457598	1215878	irradiated
SP10951	Alfalfa	I	1259310	1193463	irradiated
SP10954	Guarana	I	6624	7017	irradiated
SP10895	Cinnamon	B	363	359	not irradiated
SP10897	Cumin	B	456	552	not irradiated
SP10902	Thyme	B	1656	997	
SP10951	Alfalfa	B	3725	1627	
SP10952	Green tea	B	606	537	
SP10954	Guarana	B	914	779	

Calibrated

			Lab 24	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	540	6435
SP10897	Cumin	U	23643	19490
SP10902	Thyme	U	663769	691861
SP10950	Ginseng	U	552748	654805
SP10951	Alfalfa	U	800040	573404
SP10954	Guarana	U	5253	3014
SP10895	Cinnamon	I	1300	1578
SP10897	Cumin	I	30480	31983
SP10902	Thyme	I	1146106	1439974
SP10950	Ginseng	I	887745	807605
SP10951	Alfalfa	I	909905	942432
SP10954	Guarana	I	18567	9371
SP10895	Cinnamon	B	718	1111
SP10897	Cumin	B	21312	23438
SP10902	Thyme	B	701783	733326
SP10951	Alfalfa	B	660005	702909
SP10952	Green tea	B	20428	15102
SP10954	Guarana	B	4385	4383

Table C.15 PSL screening and Cal PSL data for lab 24

			Lab 25		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	356	348	not irradiated
SP10897	Cumin	U	276	270	not irradiated
SP10902	Thyme	U	726	295	not irradiated
SP10950	Ginseng	U	38718	36917	irradiated
SP10951	Alfalfa	U	1794	1343	not irradiated
SP10954	Guarana	U	514	511	not irradiated
SP10895	Cinnamon	I	1399	1251	not irradiated
SP10897	Cumin	I	34699	37761	irradiated
SP10902	Thyme	I	1691464	1744061	irradiated
SP10950	Ginseng	I	1709919	1255448	irradiated
SP10951	Alfalfa	I	1401278	1302744	irradiated
SP10954	Guarana	I	11529	6651	irradiated
SP10895	Cinnamon	B	355	463	not irradiated
SP10897	Cumin	B	1557	684	not irradiated
SP10902	Thyme	B	3602	1582	not irradiated
SP10951	Alfalfa	B	3073	4606	not irradiated
SP10952	Green tea	B	1808	585	not irradiated
SP10954	Guarana	B	679	770	not irradiated

Calibrated

			Lab 25	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	1108	2357
SP10897	Cumin	U	20671	11564
SP10902	Thyme	U	525135	551719
SP10950	Ginseng	U	382677	312382
SP10951	Alfalfa	U	617280	619608
SP10954	Guarana	U	3674	2584
SP10895	Cinnamon	I	962	890
SP10897	Cumin	I	17088	16765
SP10902	Thyme	I	1125188	903418
SP10950	Ginseng	I	1105347	887113
SP10951	Alfalfa	I	769937	696813
SP10954	Guarana	I	6825	3567
SP10895	Cinnamon	B	39418	32236
SP10897	Cumin	B	16596	15525
SP10902	Thyme	B	515418	474763
SP10951	Alfalfa	B	461291	488601
SP10952	Green tea	B	7734	7323
SP10954	Guarana	B	4110	4230

Table C.16 PSL screening and Cal PSL data for lab 25

			Lab 26	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	372	216
SP10897	Cumin	U	342	181
SP10902	Thyme	U	388	498
SP10950	Ginseng	U	40778	40177
SP10951	Alfalfa	U	1263	981
SP10954	Guarana	U	814	468
SP10895	Cinnamon	I	4205	879
SP10897	Cumin	I	77396	23641
SP10902	Thyme	I	1260489	1320280
SP10950	Ginseng	I	1062332	879164
SP10951	Alfalfa	I	977079	888417
SP10954	Guarana	I	14478	11099
SP10895	Cinnamon	B	355	362
SP10897	Cumin	B	644	643
SP10902	Thyme	B	2673	1439
SP10951	Alfalfa	B	2282	859
SP10952	Green tea	B	832	859
SP10954	Guarana	B	551	1187

Table C.17 PSL screening data for lab 26

			Lab 28		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	375	371	Negative
SP10897	Cumin	U	328	310	Negative
SP10902	Thyme	U	452	462	Negative
SP10950	Ginseng	U	38755	38495	Positive
SP10951	Alfalfa	U	1129	1351	Intermediate
SP10954	Guarana	U	574	486	Negative
SP10895	Cinnamon	I	1396	1005	Intermediate
SP10897	Cumin	I	48208	44635	Positive
SP10902	Thyme	I	1640819	1698644	Positive
SP10950	Ginseng	I	1274805	1419613	Positive
SP10951	Alfalfa	I	1492617	1570181	Positive
SP10954	Guarana	I	10579	10547	Positive
SP10895	Cinnamon	B	334	321	Negative
SP10897	Cumin	B	1148	936	Intermediate
SP10902	Thyme	B	1722	1481	Intermediate
SP10951	Alfalfa	B	1822	2916	Intermediate
SP10952	Green tea	B	525	522	Negative
SP10954	Guarana	B	892	990	Intermediate

Calibrated

			Lab 28	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	1184	995
SP10897	Cumin	U	21012	18630
SP10902	Thyme	U	802583	812151
SP10950	Ginseng	U	673247	544330
SP10951	Alfalfa	U	482504	445308
SP10954	Guarana	U	7806	9754
SP10895	Cinnamon	I	1505	1207
SP10897	Cumin	I	38382	29725
SP10902	Thyme	I	1247665	1285584
SP10950	Ginseng	I	792513	750701
SP10951	Alfalfa	I	877338	745585
SP10954	Guarana	I	10980	11008
SP10895	Cinnamon	B	994	1279
SP10897	Cumin	B	21086	21597
SP10902	Thyme	B	735432	732891
SP10951	Alfalfa	B	434663	477636
SP10952	Green tea	B	8980	9836
SP10954	Guarana	B	5409	4662

Table C.18 PSL screening and Cal PSL data for lab 28

			Lab 29	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	1323588	1295669
SP10897	Cumin	U	2043991	3012998
SP10902	Thyme	U	290	311
SP10950	Ginseng	U	2269	3248
SP10951	Alfalfa	U	1523	1479
SP10954	Guarana	U	8321	9221
SP10895	Cinnamon	I	309	256
SP10897	Cumin	I	87853	70673
SP10902	Thyme	I	289	321
SP10950	Ginseng	I	1263	2421
SP10951	Alfalfa	I	1406923	1578924
SP10954	Guarana	I	1155	925
SP10895	Cinnamon	B	452	501
SP10897	Cumin	B	534	322
SP10902	Thyme	B	7148	6128
SP10951	Alfalfa	B	366	424
SP10952	Green tea	B	32680	47401
SP10954	Guarana	B	1852	1899

			Lab 30	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	848	699
SP10897	Cumin	U	1021	928
SP10902	Thyme	U	651	660
SP10950	Ginseng	U	79974	88962
SP10951	Alfalfa	U	1748	1615
SP10954	Guarana	U	682	718
SP10895	Cinnamon	I	2276	1773
SP10897	Cumin	I	11621	51373
SP10902	Thyme	I	2009097	1884255
SP10950	Ginseng	I	1816847	1604716
SP10951	Alfalfa	I	1744935	1578900
SP10954	Guarana	I	17287	18351
SP10895	Cinnamon	B	583	339
SP10897	Cumin	B	852	1009
SP10902	Thyme	B	2642	1393
SP10951	Alfalfa	B	5883	7042
SP10952	Green tea	B	589	1641
SP10954	Guarana	B	2261	2670

Table C.19 PSL screening data for lab 29 and lab 30

			Lab 31		
Sample	Description	Status	Pot 1	Pot 2	**Evaluation**
SP10895	Cinnamon	U	375	223	positive
SP10897	Cumin	U	480	460	positive
SP10902	Thyme	U	548	586	negative
SP10950	Ginseng	U	48757	49369	intermediate
SP10951	Alfalfa	U	1257	1625	intermediate
SP10954	Guarana	U	922	745	intermediate
SP10895	Cinnamon	I	1685	1311	negative
SP10897	Cumin	I	51735	61486	positive
SP10902	Thyme	I	2673712	2596048	negative
SP10950	Ginseng	I	2192932	2325025	intermediate
SP10951	Alfalfa	I	2191720	2346474	positive
SP10954	Guarana	I	16538	8719	intermediate
SP10895	Cinnamon	B	430	346	negative
SP10897	Cumin	B	1370	2749	intermediate
SP10902	Thyme	B	22804	10659	positive
SP10951	Alfalfa	B	3696	4010	negative
SP10952	Green tea	B	498	464	positive
SP10954	Guarana	B	2570	1153	positive

Calibrated

			Lab 31	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	2050	2784
SP10897	Cumin	U	33727	36780
SP10902	Thyme	U	1076379	1129807
SP10950	Ginseng	U	867202	758107
SP10951	Alfalfa	U	781410	891834
SP10954	Guarana	U	13571	11218
SP10895	Cinnamon	I	2761	2530
SP10897	Cumin	I	38375	42087
SP10902	Thyme	I	2051582	2009339
SP10950	Ginseng	I	1515929	1485889
SP10951	Alfalfa	I	1514627	1579120
SP10954	Guarana	I	17009	9007
SP10895	Cinnamon	B	1810	1751
SP10897	Cumin	B	29515	28674
SP10902	Thyme	B	1140272	1135758
SP10951	Alfalfa	B	1122685	1112228
SP10952	Green tea	B	24174	25735
SP10954	Guarana	B	30856	6663

Table C.20 PSL screening and Cal PSL data for lab 31

			Lab 32		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	446	408	untreated
SP10897	Cumin	U	552	693	untreated
SP10902	Thyme	U	533	540	untreated
SP10950	Ginseng	U	78081	85405	irradiated
SP10951	Alfalfa	U	1231	1712	mixtures with irradiated materials
SP10954	Guarana	U	683	553	untreated
SP10895	Cinnamon	I	2518	1858	mixtures with irradiated materials
SP10897	Cumin	I	64211	54490	irradiated
SP10902	Thyme	I	2774638	2559089	irradiated
SP10950	Ginseng	I	1084008	1982898	irradiated
SP10951	Alfalfa	I	2118995	1738688	irradiated
SP10954	Guarana	I	11005	20717	irradiated
SP10895	Cinnamon	B	399	518	untreated
SP10897	Cumin	B	1123	984	mixtures with irradiated materials
SP10902	Thyme	B	6782	5346	mixtures with irradiated materials
SP10951	Alfalfa	B	2544	3026	mixtures with irradiated materials
SP10952	Green tea	B	615	520	untreated
SP10954	Guarana	B	2429	1733	mixtures with irradiated materials

			Lab 33		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	121	171	Negative
SP10897	Cumin	U	400	309	Negative
SP10902	Thyme	U	494	282	Negative
SP10950	Ginseng	U	70862	54909	Positive
SP10951	Alfalfa	U	2746	3588	Intermediate
SP10954	Guarana	U	551	480	Negative
SP10895	Cinnamon	I	1592	938	Intermediate
SP10897	Cumin	I	34562	36356	Positive
SP10902	Thyme	I	1940698	2029268	Positive
SP10950	Ginseng	I	1878093	1987929	Positive
SP10951	Alfalfa	I	1655855	1139999	Positive
SP10954	Guarana	I	8677	11830	Positive
SP10895	Cinnamon	B	346	312	Negative
SP10897	Cumin	B	369	1333	
SP10902	Thyme	B	2382	2654	Intermediate
SP10951	Alfalfa	B	3219	3521	Intermediate
SP10952	Green tea	B	509	1794	
SP10954	Guarana	B	787	1645	Intermediate

Table C.21 PSL screening data for lab 32 and lab 33

			Lab 34		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	508	549	unclassified
SP10897	Cumin	U	890	946	unclassified
SP10902	Thyme	U	471	414	unclassified
SP10950	Ginseng	U	23439	30486	irradiated
SP10951	Alfalfa	U	1181	920	unclassified
SP10954	Guarana	U	499	560	unclassified
SP10895	Cinnamon	I	1549	1521	unclassified
SP10897	Cumin	I	15479	21058	irradiated
SP10902	Thyme	I	767338	860987	irradiated
SP10950	Ginseng	I	645583	842308	irradiated
SP10951	Alfalfa	I	354972	575813	irradiated
SP10954	Guarana	I	5374	5983	irradiated
SP10895	Cinnamon	B	590	549	unclassified
SP10897	Cumin	B	797	1072	unclassified
SP10902	Thyme	B	734	850	unclassified
SP10951	Alfalfa	B	1742	2492	unclassified
SP10952	Green tea	B	703	593	unclassified
SP10954	Guarana	B	820	1419	unclassified

Calibrated

			Lab 34		
Sample	Description	Status	Pot 1	Pot 2	Evaluation after Calibration
SP10895	Cinnamon	U	952	720	not irradiated
SP10897	Cumin	U	16890	19740	not mesurable by PSL
SP10902	Thyme	U	700061	657419	not irradiated
SP10950	Ginseng	U	526407	443370	irradiated
SP10951	Alfalfa	U	330888	124431	not mesurable by PSL
SP10954	Guarana	U	3137	3163	not irradiated
SP10895	Cinnamon	I	1385	1164	irradiated
SP10897	Cumin	I	27371	25247	irradiated
SP10902	Thyme	I	1270717	1227431	irradiated
SP10950	Ginseng	I	458058	686858	irradiated
SP10951	Alfalfa	I	267293	643021	irradiated
SP10954	Guarana	I	11428	7111	irradiated
SP10895	Cinnamon	B	956	1024	not irradiated
SP10897	Cumin	B	15065	21752	not mesurable by PSL
SP10902	Thyme	B	509585	581954	irradiated or not mesurable by PSL
SP10951	Alfalfa	B	508481	480137	not mesurable by PSL
SP10952	Green tea	B	7099	6973	not irradiated
SP10954	Guarana	B	3711	4979	irradiated

Table C.22 PSL screening and Cal PSL data for lab 34

			Lab 35		
Sample	Description	Status	Pot 1	Pot 2	Evaluation
SP10895	Cinnamon	U	418	415	non irradiated
SP10897	Cumin	U	484	333	non irradiated
SP10902	Thyme	U	476	627	non irradiated
SP10950	Ginseng	U	54054	64749	irradiated
SP10951	Alfalfa	U	978	1275	inconclusive
SP10954	Guarana	U	791	664	inconclusive
SP10895	Cinnamon	I	1576	2037	inconclusive
SP10897	Cumin	I	66549	60801	irradiated
SP10902	Thyme	I	2231959	2393347	irradiated
SP10950	Ginseng	I	1044029	981440	irradiated
SP10951	Alfalfa	I	1267192	1040826	irradiated
SP10954	Guarana	I	9278	12107	irradiated
SP10895	Cinnamon	B	400	634	non irradiated
SP10897	Cumin	B	900	1058	inconclusive
SP10902	Thyme	B	2249	2058	inconclusive
SP10951	Alfalfa	B	3419	2407	inconclusive
SP10952	Green tea	B	966	549	inconclusive
SP10954	Guarana	B	1677	1724	inconclusive

Calibrated

			Lab 35		
Sample	Description	Status	Pot 1	Pot 2	Evaluation after Calibration
SP10895	Cinnamon	U	923	1902	non irradiated
SP10897	Cumin	U	36148	47927	non irradiated
SP10902	Thyme	U	1308107	1372369	non irradiated
SP10950	Ginseng	U	698026	1118483	irradiated component
SP10951	Alfalfa	U	604717	688817	irradiated component
SP10954	Guarana	U	5901	6146	irradiated component
SP10895	Cinnamon	I	2698	2423	irradiated
SP10897	Cumin	I	51917	41131	irradiated
SP10902	Thyme	I	1973889	2193028	irradiated
SP10950	Ginseng	I	959722	818493	irradiated
SP10951	Alfalfa	I	861417	942568	irradiated
SP10954	Guarana	I	12307	9143	irradiated
SP10895	Cinnamon	B	955	1256	non irradiated
SP10897	Cumin	B	37249	31299	irradiated component
SP10902	Thyme	B	1474805	1412820	non irradiated
SP10951	Alfalfa	B	791617	868202	irradiated component
SP10952	Green tea	B	16507	18345	non irradiated
SP10954	Guarana	B	13790	14172	irradiated component

Table C.23 PSL screening and Cal PSL data for lab 35

			Lab 36	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	431	298
SP10897	Cumin	U	271	221
SP10902	Thyme	U	409	664
SP10950	Ginseng	U	112712	80019
SP10951	Alfalfa	U	1738	1841
SP10954	Guarana	U	566	634
SP10895	Cinnamon	I	2749	1624
SP10897	Cumin	I	77972	52821
SP10902	Thyme	I	1930855	2179845
SP10950	Ginseng	I	1507841	1553475
SP10951	Alfalfa	I	1928570	1916682
SP10954	Guarana	I	14310	15144
SP10895	Cinnamon	B	409	596
SP10897	Cumin	B	788	1246
SP10902	Thyme	B	3606	3224
SP10951	Alfalfa	B	3244	11708
SP10952	Green tea	B	1327	1177
SP10954	Guarana	B	1555	3659

			Lab 37	
Sample	Description	Status	Pot 1	Pot 2
SP10895	Cinnamon	U	792	709
SP10897	Cumin	U	959	425
SP10902	Thyme	U	613	520
SP10950	Ginseng	U	75096	66608
SP10951	Alfalfa	U	2125	1710
SP10954	Guarana	U	1322	735
SP10895	Cinnamon	I	21895	35925
SP10897	Cumin	I	66087	63392
SP10902	Thyme	I	2800616	2759221
SP10950	Ginseng	I	2500235	2024877
SP10951	Alfalfa	I	1759100	2362371
SP10954	Guarana	I	24161	15104
SP10895	Cinnamon	B	537	543
SP10897	Cumin	B	1053	648
SP10902	Thyme	B	1938	1915
SP10951	Alfalfa	B	5020	5663
SP10952	Green tea	B	849	1151
SP10954	Guarana	B	2993	2937

Table C.24 PSL screening data for lab 36 and lab 37

APPENDIX D – PARTICIPANTS' RAW TL DATA

Lab No.	1									
TL reader type	Harshaw M 3500									
Units for intensity	nC									
MDL	0.13412634	0.11110348								
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	Evaluation	Comments
1	36.27	26.46	2.047308312	small peak in relevant temperature range	1563	571	2.88676236	distinct peak in relevant temperature range	positive	
2	371.2	258.2	1.65914397	distinct peak in relevant temperature range					positive	
3	15.74	51.54	0.0112614	no distinct peak in relevant temperature range					negative	
4	22.65	198.6	0.00426593	no distinct peak in relevant temperature range					positive	negative in water
5	26.69	20.45	1.81954887	small peak in relevant temperature range					positive	
6										
7										
8	29.37	29.39	4.01353638	small peak in relevant temperature range					positive	
9	22.17	47.32	0.03752288	no distinct peak in relevant temperature range					positive	negative in water
10	19.24	60.87	0.00980769	no distinct peak in relevant temperature range					positive	negative in water
11	137.3	88.19	1.89670223	distinct peak in relevant temperature range	44.09	36.64	2.03708251	small peak in relevant temperature range	positive	
12										
13										
14	26.08	51.18	0.00692457	no distinct peak in relevant temperature range					negative	
15	26.7	21.62	2.37381275	small peak in relevant temperature range					positive	
16	28.88	233.6	0.00200853	no distinct peak in relevant temperature range					positive	negative in water
17	55.04	432.3	0.03951694	small peak in relevant temperature range					positive	negative in water
18	21.13	45.11	0.04295796	no distinct peak in relevant temperature range					positive	negative in water

Table D.1 TL data for TL Laboratory 1

Lab No.	2								
TL reader type	Risö TL/OSL-DA15								
Units for intensity	Counts								
MDL	499 counts (11.04.2007)								
(full temperture interval I)	516 counts (16.04.2007)								
Sample data									
	Aliquot A (Intensity of full temperature interval I)				Aliquot B ((Intensity of full temperature interval I)				
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensity	G2 Intensit	G1/G2	G1 peak shape	Evaluation
1									
2	3867166	6305463	0.61	broad dominant peak, maximum in temperature interval I	8046202	474906	1.69	broad dominant peak, maximum in temperature	irradiated
3	1167	8803877	0.0001	no peak in temperature interval I	1101	1709176	0.0006	no peak in temperature	not irradiated
4									
5									
6	1812	460369	0.004	no peak in temperature interval I	1136	916922	0.001	no peak in temperature	not irradiated
7									
8	1447688	848579	1.71	broad dominant peak, maximum in temperature interval I	6108679	2281638	2.68	dominant peak, maximum in temperature interval I with shoulder at lower	irradiated
9									
10									
11									
12	715273	1269952	0.56	broad dominant peak, maximum in temperature interval I	1930487	2258681	0.85	broad dominant peak, maximum in temperature	irradiated
13									
14	13998	2184814	0.006	no peak in temperature interval I	2462	761582	0.003	no peak in temperature	not irradiated
15	10915000	3813689	2.86	broad dominant peak, maximum in temperature interval I, wtih shoulder at lower temperature	18817265	4916720	3.83	dominant peak, maximum in temperature interval I, with shoulder at lower temperature	irradiated
16	2508	1852915	0.001	no peak in temperature interval I	2080	3774502	0.0006	no peak in temperature	not irradiated
17									
18	900	1991134	0.0005	no peak in temperature interval I	2134	4584025	0.0005	no peak in temperature	not irradiated

Table D.2 TL data for TL Laboratory 2

Lab No.	3									
TL reader type	**Harshaw 3500**									
nits for intensi	**nC**									
MDL	**0,620 nC**									
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	Evaluation	Comments
1										
2	40540	19729	2.054843	peak at 199 °C	48154	22772	2.114614	peak at 206 °C	Irradiated	
3	3.757	100.3	0.037458	peak at 197 °C	5.287	175.3	0.03016	peak at 202 °C	mixture	
4										
5										
6	7.781	121.7	0.063936	peak at 206 °C	7.28	65.62	0.110942	peak at 208 °C	mixture	
7										
8	333.4	53.75	6.202791	peak at 223 °C	751.9	220.2	3.414623	peak at 208 °C	Irradiated	
9										
10										
11										
12	49.05	120.5	0.407054	peak at 230 °C	44.84	81.81	0.548099	peak at 230 °C	mixture	
13										
14	1.253	140.5	0.008918	peak at 316 °C	1.197	206.5	0.005797	peak at 316 °C	untreated	
15	2187	182.2	12.00329	peak at 235 °C	1019	178.5	5.708683	peak at 235 °C	Irradiated	
16	7.123	25219	0.000282	T>200 °C	4.898	19810	0.000247	T>200 °C	untreated	geological signal
17										
18	58.73	15562	0.003774	peak at 202 °C	75.61	15836	0.004775	peak at 201 °C	mixture	

Table D.3 TL data for TL Laboratory 3

Lab No.		TL Lab 4											
L reader type		Harshaw TLD-Reader 3500											
its for intensity		nano Coulomb (nC)											
MDL		20 nC											
Sample data													
	Aliquot A				**Aliquot B***								
Sample	G1 Intensit	G2 Intensit	G1/G2	G1 peak sh	G1 Intensit	G2 Intensit	G1/G2	G1 peak sh	Evaluation	Comments			
1													
2	2952	977	3.02	gaussian, 200°C max					irradiated				
3	6.23	7.78	0.80	gaussian, 200°C max					untreated	Inensity G1 under MDL			
4													
5													
6	2.33	4.46	0.52	very small, 195°Cmax					untreated	Inensity G1 under MDL			
7													
8	224.9	40.1	5.61	not ideal gaussian, 240°C max					irradiated	**			
9													
10													
11													
12	31.7	34.3	0.92	gaussian, 240°C max					irradiated	**			
13													
14	2.72	18.39	0.15	very small					untreated	both inensities G1and G2 under MDL			
15	323	340	??	gaussian, 230°C max					irradiated	***			
16	23.8	2638	0.01	not gaussian, 200°C max					untreated	Inensity G1 under MDL			
17													
18	99.5	3626	0.03	gaussian, 200°C max					mixture				
					*	**Aliquot B wasnot possible to get**							
					**	**curves are not ideally gaussian, organic backround?**							
					***	**only glow curve in the 1. TL, not after irradiation, what does it means??**							

Table D.4 TL data for TL Laboratory 4

Lab No.	6						
TL reader type	No 194-05/2005-b						
Units for intensity	counts						
MDL	1693						
Sample data							
	Aliquot A				Aliquot B		
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensity	G2 Intensity	G1/G2
1	64 248 510	40 471 176	1.59	rising from 130°CMax at 190°C	40 587 265	23 874 479	1.7
2	24 199 302	15 769 064	1.54	rising from 120°CMaximum at 185-	86 095 808	50 999 908	1.69
3	2 152	7 037 403	< 0,1	rising baseline from 160 °C	29 287	8 039 827	< 0,1
4	535 674	82787831	0.006	rising from 130°Cfirst Max at 190°CMin at	378 834	84318781	0.004
5	2 052 302	1 141 903	1.8	rising from 135°CMax at 210°C shoulder at	830 712	372 824	2.23
6	7 511	2 291 436	< 0,1	rising from 120°CMax at 185°C	31 203	2440992	< 0,1
7	130 078	311 217	0.42	rising from 130°C Max at 200°C small shoulder	891522	1 021 398	0.87
8	10 616 024	5 760 671	1.84	strong rising from 120°C Max at 180°C	15 614 008	9 561 232	1.63
9	18 071	9 195 877	< 0,1	riseing from 170°C small shoulder at	85 352	9 905 079	< 0,1
10	17 823	15 576 384	< 0,1	rising from 200°Cto 280°C, shoulder, slow	5 677	5 696 940	< 0,1
11	38 028 596	22 797 741	1.67	rising from 130°CMax at 195°C	29 864 554	17 679 256	1.69
12	2 091 221	3 621 818	0.58	rising from 140°CMax at 225°CMin at	3 719 166	7 927 880	0.47
13	3 626	824 836	< 0,1	rising from 220°CMax at 310°C	2 632	945 156	< 0,1
14	77 391	11 143 938	< 0,1	slow rising from 200°Cto Max at 350°C	29 279	7 970 959	< 0,1
15	49 981 583	12 680 462	3.94	rising from 140°Cshoulder at 175°CMax at	22 609 117	5 643 695	4
16	69 076	109 481 147	< 0,1	from 180°C slow rising to Max 370°C	65 090	102 343 715	< 0,1
17	6 964 666	122 393 440	0.057	rising from 160°CMax at 235°Csmall shoulder	3141701	48410961	0.065
18	125 362	38 798 119	0.003	rising from 140°CMax at 185°CMin at	171 100	37 878 138	0.005

Table D.5 TL data for TL Laboratory 6

Lab No.	7									
TL reader type	TLD 3500									
Units for intensit	nC									
MDL	3,12nC									
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	Evaluation	Comments
1	9953.00	3740.00	2.66	peak in ROI1	7709.00	1839.00	4.19	peak in ROI1	irradiated	
2	32.11	13.20	2.43	peak in ROI1	19.57	3.31	5.92	peak in ROI1	irradiated	
3	1.29	17.73	0.07	no peak inROI 1	1.36	11.71	0.12	no peak inROI 1	untreated	
4	3.17	32.87	0.10	little peak in ROI1	33.37	219.00	0.15	peak in ROI1	irradiated	mixtures containing irradiated materials
5	6.26	5.19	1.21	small peak higher temp.	10.72	8.84	1.21	small peak higher temp.	irradiated	difficult
6	1.20	9.71	0.12	little peak in ROI1	1.82	4.57	0.40	little peak in ROI1	irradiated	mixtures containing irradiated materials
7	9.26	5.42	1.71	little peak in ROI1	0.72	0.53	1.37	peak in ROI1	irradiated	inhomogen sample
8	55.41	23.86	2.32	peak in ROI1	32.59	8.359	3.90	peak in ROI1	irradiated	
9	7.338	71.27	0.10	small peak higher temp.	1.959	4.12	0.48	breadth peak in ROI1	irradiated	difficult
10	3.903	100.1	0.04	breadth peak in ROI1	6.371	160.8	0.04	breadth peak in ROI1	irradiated	difficult
11	791.1	349.6	2.26	peak in ROI1	833.2	387.5	2.15	peak in ROI1	irradiated	
12	1.158	1.558	0.74	small peak higher temp.	1.048	0.9698	1.08	small peak higher temp.	irradiated	inhomogen sample
13	0.686	4.33	0.16	no peak	0.6246	0.928	0.67	no peak	untreated	
14	1.083	2.665	0.41	no peak	0.7171	7.683	0.09	no peak	untreated	
15	40.51	24.27	1.67	small peak higher temp.	36.01	53.55	0.67	small peak higher temp.	irradiated	difficult
16	0.3199	1.972	0.16	no peak	0.3461	51.52	0.01	no peak	untreated	
17	11.68	2165	0.01	no peak inROI 1	51.52	803.7	0.06	no peak inROI 1	untreated	
18	0.3265	17.17	0.02	no peak inROI 1	0.2276	6.559	0.03	no peak inROI 1	untreated	

Table D.6 TL data for TL Laboratory 7

Lab No.	8								
TL reader type	TLD 3500								
Units for intensity	nC								
MDL	0.899								
Sample data									
	Aliquot A				Aliquot B				
Sample	G1 Intensit	G2 Intensit	G1/G2	G1 peak sh	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	Evaluation
1	24950	10170	2.45		16380	14160	1.16		irradiated
2	2312	1017	2.27		3211	1741	1.84		irradiated
3	0.3147	159.7	0.002		1.486	56.46	0.026		untreated
4	23.42	2328	0.010		4.796	4288	0.001		mixtures containing irratiated materials
5	437.5	203.2	2.15		32.92	15.69	2.10		irradiated
6	0.4533	73.66	0.006		0.9674	328.6	0.003	small peak (ROI 1)	mixtures containing irratiated materials
7	0.2266	7.02	0.032		0.4269	0.7136	0.60		untreated
8	66.26	36.42	1.82		47.32	38.56	1.23		irradiated
9	53.59	1408	0.038		70.62	1800	0.039		mixtures containing irratiated materials
10	3.387	1766	0.002		6.601	10210	0.001		untreated
11	7689	5644	1.36		4300	3237	1.33		irradiated
12	791.8	1534	0.52		114.3	287.4	0.40		mixtures containing irratiated materials
13	0.6519	0.7393	0.88		0.3759	0.9911	0.38		untreated
14	101.8	215.8	0.47		1.582	610.4	0.003		mixtures containing irratiated materials
15	110.2	31.08	3.55		3065	537.6	5.70		irradiated
16	0.8136	378.9	0.002		0.4349	501.7	0.001		untreated
17	182.5	5147	0.035		182.9	15240	0.012		mixtures containing irratiated materials
18	1.203	159.1	0.008		0.8775	1046	0.001		untreated

Table D.7 TL data for TL Laboratory 8

Lab No.	9								
TL reader type	Harshaw QS 3500								
Units for intensity	nC								
MDL	1.63								
Sample data									
	Aliquot A				Aliquot B				
Sample	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	Evaluation
1	32912	9787.7	3.363	Peak at 181 °C	33546	3634.4	9.230	Peak at 181 °C	Irradiated
2	15223	3058.3	4.978	Peak at 174 °C	20744	4077.1	5.088	Peak at 174 °C	Irradiated
3	0.40288	454.76	0.001	shoulder at 340 °C	0.32662	503.06	0.001	shoulder at 340 °C	untreated
4	15.005	3673	0.004	shoulder at 267, Peak at 340	55.29	7700	0.007	peak at 340 oC	untreated
5	2070.5	411.17	5.036	Peak at 194 °C	1985.8	552.1	3.597	Peak at 186 °C	Irradiated
6	25.422	556.12	0.046	peak at 170 °C	34.38	508.85	0.068	peak at 175 °C	mixture
7	31.326	175.03	0.179	Peak at 187 °C	13.47	296.12	0.045	Shoulder at 187°C?peak at 274 °C	mixture
8	1596.3	554.67	2.878	Peak at 177 °C	1886	622.3	3.031	Peak at 177 °C	Irradiated
9	50.663	2514.2	0.020	Peak at 209°C?and 347°C	75.622	2593.2	0.029	Peak at 208°C?and 351°C	mixture
10	4.3177	2801.5	0.002	Peak at 267 °C and 334 °C	10.465	7394.2	0.001	Peak at 264 °C and 340 °C	untreated
11	13523	3666	3.689	Peak at 181 °C	17932	4389.2	4.085	Peak at 188 °C	Irradiated
12	276.4	451.66	0.612	Peak at 208 °C and 335 °C	1056.1	768.88	1.374	Peak at 208 °C and 337°C	Irradiated
13	0.9215	175.93	0.005	small peak at 288 °C	0.31825	47.208	0.007	small peak at 279 °C	untreated
14	5.7006	1316.9	0.004	Peak at 347 °C	5.7445	625.54	0.009	shoulder at 249 °C?peak at 345°C	untreated
15	12047	1625.9	7.409	Peak at 211°C and 334 °C	11599	1408.1	8.237	Peak at 209°C and 337 °C	Irradiated
16	3.5116	4527.7	0.001	(peak at 340 °C)	2.7534	3326.6	0.001	(peak at 340 °C)	untreated
17	1076.9	18670	0.058	Peak at 211°C	1017.6	15747	0.065	Peak at 210°C	mixture
18	37.487	3868.4	0.010	Peak at 177 °C	19.86	3749.3	0.005	shoulder at 177?°C	mixture

Table D.8 TL data for TL Laboratory 9

Lab No.	11									
TL reader type	Riso TL/OSL									
Units for intensity	AU									
MDL	489									
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	Evaluation	Comments
1										
2	37461	37801	0.991	Max ≅ 170°C	51375	42209	1.217	Max ≅ 190°C		irradiated
3	121	1775	0.068	Background	521	1792	0.291	Max ≅ 200°C		non irradiated
4										
5										
6	130	4388	0.030	Background	115	840	0.137	Background		non irradiated
7										
8	7347	2757	2.665	Max ≅ 160°C	20709	21431	0.966	Bimodal		irradiated
9										
10										
11										
12	2091	16615	0.126	Bimodal	4108	8357	0.492	Bimodal		might contain irradiated components
13										
14	600	31506	0.019	Max > 300°C	290	19898	0.015	Max > 300°C		non irradiated
15	63503	18828	3.373	Max ≅ 230°C	41701	7681	5.429	Max ≅ 220°C		irradiated
16	1536	170585	0.009	Bimodal	136	93997	0.001	Max > 300°C		non irradiated
17										
18	236	62495	0.004	Max > 300°C	251	109835	0.002	Max > 300°C		non irradiated

Table D.9 TL data for TL Laboratory 11

Lab No.	**12**									
TL reader type	Harshaw 3500						**LIF Data**			
nits for intensi	nC							Peak V temperature/°C		
MDL	Average x	0.0582						Chip 1	245 °C	
	Standard de	0.0214						Chip 2	248 °C	
	MDL (x + 3s	0.1223								
	10*MDL	1.223						**Mean**	**247 °C**	
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensity	G2 Intensit	G1/G2	G1 peak shape	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	Evaluation	Comments
1	18570	12200	1.52	Max. in Interval I; 240 °C	24370	16280	1.50	Max. in Interval I; 225 °C	irradiated	
1	21020	16650	1.26	Max. in Interval I; 240 °C	33760	11890	2.84	Max. in Interval I; 220 °C	irradiated	
2	5175	2351	2.20	Max. in Interval I; 200 °C					irradiated	
3	2.328	176.4	0.01	no Max. in Interval I	2.763	133.6	0.02	no Max. in Interval I	not irradiated	
4	62.24	6846	0.01	first Max.in Interval I; 200 °C; second Max 300 °C					irradiated (probably mixture)	
5	433.7	124.3	3.49	Max. in Interval I; 225 °C					irradiated	
6	1.076	140.3	0.01	no Max. in Interval I	1.784	44.94	0.04	no Max. in Interval I	not irradiated	
7	0.0318	0.3316	0.10	no Max. in Interval I					<10 MDL	not enough minerals
8	574.2	180.1	3.19	Max. in Interval I; 220 °C	193.9	59.83	3.24	Max. in Interval I; 210 °C	irradiated	
8	206	72.39	2.85	Max. in Interval I; 210 °C	179.2	60.36	2.97	Max. in Interval I; 210 °C	irradiated	
9	84.01	2364	0.04	Max. in Interval I; 250 °C					irradiated (probably mixture)	
10	8.561	2622	0.00	no Max. in Interval I					not irradiated	wide peak
11	7200	2047	3.52	Max. in Interval I; 225 °C	5951	1202	4.95	Max. in Interval I; 225 °C	irradiated	
11	5687	1367	4.16	Max. in Interval I; 230 °C					irradiated	
12	471	48.8	9.65	Max. in Interval I; 250 °C	343.9	272.9	1.26	Max. in Interval I; 250 °C	irradiated	
12	99.38	155.1	0.64	Max. in Interval I; 260 °C	28.39	85.59	0.33	Max. in Interval I; 250 °C	irradiated	
13	21.46	14.59	1.47	Max. in Interval I; 200 °C					irradiated	
14	1.839	322.9	0.01	no Max. in Interval I	3.994	654	0.01	no Max. in Interval I	not irradiated	
15	4704	1213	3.88	Max. in Interval I; 240 °C	1427	275.6	5.18	Max. in Interval I; 240 °C	irradiated	
16	0.3902	840	0.00	no Max. in Interval I					not irradiated	
17	261.3	10040	0.03	Max. in Interval I; 260 °C	220.2	13190	0.02	Max. in Interval I; 275 °C	irradiated (probably mixture)	
17	304.2	9434	0.03	Max. in Interval I; 260 °C	251.4	10440	0.02	Max. in Interval I; 250 °C	irradiated (probably mixture)	
18	14.28	2753	0.01	no Max. in Interval I					not irradiated	mixture ????

Table D.10 TL data for TL Laboratory 12

Lab No.	13									
TL reader type	TL-DA-10									
Units for intensity	counts									
MDL (200-250°C)	177									
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	Evaluation	Comments
1										
2	1305071	1025167	1.273	max 188°C	868755	421293	2.062	max 192°C	irradiated	
3	232	1599215	0.000145	max 369°C	234	761340	0.000307	max 498°C	unirradiated	
4										
5										
6	16279	1691746	0.00962	max 180°C	5313	4855276	0.00109	max 314°C	light mixture	base on form of spectrum A
7										
8	1268781	572367	2.217	max 184°C	1351009	799212	1.690	max 184°C	irradiated	
9										
10										
11										
12	540082	1674686	0.322	max 218°C	340136	735506	0.462	max 220°C	mixture	base on form of spectra
13										
14	10800	871358	0.0124	max 354°C	8271	567712	0.0146	max 360°C	unirradiated	
15	7825574	2161707	3.620	max 226°C	5164415	2268159	2.277	max 220°C	irradiated	
16	1703	918193	0.00185	max 462°C	2691	1509832	0.00178	max 372°C	unirradiated	
17										
18	2688	592032	0.00454	max 468°C	754	6724645	0.000112	max 494°C	unirradiated	

Table D.11 TL data for TL Laboratory 13

Lab No.	15									
TL reader type	Harshaw 3500									
Units for intensity	nC									
MDL	0.25									
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	Evaluation	Comments
2	24620	12600	1.9540	Peak at about 150-250 ?	58250	36270	1.6060	Peak at about 150-250 ?	Positive	
3	0.1481	83.1	0.0018	No peak	0.1831	48.68	0.0038	No peak	Negative	
6	0.3984	253.5	0.0016	No peak	0.1588	13.34	0.0119	No peak	Negative	
8	95.32	45.94	2.0749	Peak at about 150-250 ?	321.8	165.3	1.9468	Peak at about 150-250 ?	Positive	
12	441	2423	0.1820	Very large peak at 210 ?	1039	2900	0.3583	Very large peak at 210 ?	Positive	
14	5.174	1939	0.0027	Weak peak at about 300 ?	2.042	650.7	0.0031	Weak peak at about 300 ?	Mixture	
15	3668	1607	2.2825	Peak at about 150-250 ?	11350	7223	1.5714	Peak at about 150-250 ?	Positive	
16	4.932	4914	0.0010	No peak	5.875	19600	0.0003	No peak	Negative	
18	23.05	8480	0.0027	Weak peak at about 150-250 ?	4.268	5917	0.0007	Weak peak at about 150-250 ?	Mixture	

Table D.12 TL data for TL Laboratory 15

Lab No.	16									
TL reader type	TL/OSL DA-20									
Units for intensity										
MDL	1819									
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensity	G2 Intensity	G1/G2	G1 peak sh	Evaluation	Comments
1	28761909	14059680	2.046	dominant	36641699	20022984	1.830	dominant	Irradiated	
2	2263080	2619110	0.864	dominant	662923	1088721	0.609	dominant	Irradiated	
3	1910	30291	0.063	absent	1884	42732	0.044	absent	Non irradiated	
4	9298	8926276	0.001	absent	14286	6091419	0.002	absent	Non irradiated	
5	35238	26213	1.344	dominant	21845	19221	1.137	dominant	Irradiated	
6	1454	39240	0.037	absent	1398	23625	0.059	absent	Non irradiated	
7	9326	28873	0.323	dominant	17693	68122	0.260	dominant	Irradiated	
8	7426	20625	0.360	dominant	12894	44840	0.288	dominant	Irradiated	
9	1984	46715	0.042	absent	1915	404149	0.005	absent	Non irradiated	
10	5020	8580917	0.001	absent	3769	6819728	0.001	absent	Non irradiated	
11	19140337	11539297	1.659	dominant	13979105	8540827	1.637	dominant	Irradiated	
12	78547	155566	0.505	dominant	12259	68336	0.179	dominant	Irradiated	
13	1678	75492	0.022	absent	1485	47903	0.031	absent	Non irradiated	
14	1607	28544	0.056	absent	1857	104414	0.018	absent	Non irradiated	
15	222513	260192	0.855	dominant	274338	263969	1.039	dominant	Irradiated	
16	1780	338899	0.005	absent	1814	2008959	0.001	absent	Non irradiated	
17	172329	16550156	0.010	absent	129346	7570274	0.017	absent	Non irradiated	
18	4397	551728	0.008	absent	1594	1486764	0.001	absent	Non irradiated	

Table D.13 TL data for TL Laboratory 16

Lab No.	17									
TL reader type	Harshaw TLD 3500									
Units for intensity	nC									
MDL	0.4405									
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	G1 Intensit	G2 Intensit	G1/G2	G1 peak shape	Evaluation	Comments
1										
2	18936	12323	1.537	one Peak in ROI max 211° C	21663	10576	2.048	one Peak in ROI max 218° C	irradiated	
3	45.787	237.79	0.193	one Peak in ROI max 225° C	5.6684	76.757	0.074	one Peak in ROI max 208° C	mixtures with irradiated materials	
4										
5										
6	7.8773	258.83	0.030	one Peak in ROI max 202° C	7.2393	171.53	0.042	one Peak in ROI max 205° C	mixtures with irradiated materials	
7										
8	546.34	207.08	2.638	one Peak in ROI max 215° C	1199	350.45	3.421	wide Peak in ROI max 251° C	irradiated	
9										
10										
11										
12	175.4	414.16	0.424	one Peak in ROI max 241° C	394.63	721.83	0.547	one Peak in ROI max 241° C	irradiated	
13										
14	13.863	1388.2	0.010	no Peak	6.4721	536.3	0.012	no Peak	untreated	
15	2371.2	840.48	2.821	one Peak in ROI max 228° C	5109.8	1417.2	3.606	one Peak in ROI max 245° C	irradiated	
16	3.8387	2877.8	0.001	no Peak	14.015	2829	0.005	one Peak in ROI max 271° C	untreated	
17										
18	33.101	3283.4	0.010	small Peak in ROI max 225°C	45.949	minerals lost			no final decission, mixture ?	

TableD.14 TL data for TL Laboratory 17

Lab No.	18									
TL reader type	RIS∅ TL/OSL System, model TL-DA-15; TLF programme: 50-500°C; 6 °C/s									
Units for intensi	counts									
MDL	250.8	integration (150 - 250°C)								
Operators:	K.Malec-Czechowska		G.Liśkiewic	M.Laubsztejn						
Sample data										
	Aliquot A				Aliquot B					
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	Evaluation	Comments
1	255601783	153106658	1.6694	max 220°C	316123097	177290174	1.7831	max 210°C	irradiated	
2	113946431	56184305	2.0281	max 210°C	141310560	86188739	1.6395	max 220°C	irradiated	
3	548	918710	0.0006	no TL peak	486	3925205	0.0001	no peak	untreated	
4	119535	60292281	0.0020	(max 300 -400°C)	142283	67396002	0.0021	max 365°C	untreated	
5	4028968	2183152	1.8455	max 240°C	717280	652609	1.0991	max 240°C	irradiated	
6	29975	674994	0.0444	max 220°C	5274	1194530	0.0044	max 225°C ?	mixture containing irradiated material	second peak G1 at 360°C (Aliquot B)
7	372477	2563200	0.1453	max 230°C	20315	502733	0.0404	max 245°C	mixture containing irradiated material	very low content
8	7151060	2851997	2.5074	max 210°C	22890180	9711842	2.3569	max 225°C	irradiated	
9	320604	23134817	0.0139	max 255°C	688641	45846612	0.0150	max 225°C	mixture containing irradiated material	second peak G1 (Aliquot A and B) at 375°C
10	71459	91107743	0.0008	(max 310-375°C)	117027	136113572	0.0009	max between 300-400°C	untreated	
11	59353363	22405055	2.6491	max 225°C	90694538	31666658	2.8640	max 215°C	irradiated	
12	7579108	12334054	0.6145	max 245°C	7306612	15333692	0.4765	max 245°C	irradiated	
13	260	1402716	0.0002	no TL peak	20046	5287353	0.0038	max 305°C	untreated	
14	112437	18090857	0.0062	(max 375°C)	57143	8744667	0.0065	max 400°C	untreated	
15	22978443	5037169	4.5618	max 240°C	2957631	469842	6.2949	max 245°C	irradiated	second peak near 360°C
16	9382	27334972	0.0003	(max 450°C)	26487	45691773	0.0006	max 400°C	untreated	
17	429761	24183932	0.0178	(max 300°C)	2131335	49394666	0.0431	max 270°C	untreated	
18	379833	57547752	0.0066	max 210°C	966020	120807839	0.0080	max 210°C	mixture containing irradiated material	

Table D.15 TL data for TL Laboratory 18

Lab No.	19								
TL reader type	Harshaw TLD 4000								
Units for intensity	nC								
MDL	0.53								
Sample data									
	Aliquot A				Aliquot B				
Sample	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	G1 Intensity	G2 Intensity	G1/G2	G1 peak shape	Evaluation
1	16871.51	5789.2	2.91	maximum 235 C	17995.44	7229, 58	2.49	maximum 235 C	irradiated
2	369.01	152.51	2.42	maximum 250 C	4967.2	2377.58	2.09	maximum 250 C	irradiated
3	0.4	14.83	0.027	maximum 450 C	0.42	8.28	0.05	maximum 450 C	untreated
4	0.71	28.02	0.025	maximum 330 C	0.67	23.72	0.028	maximum 310 C	irradiated
5	203.74	74.53	2.73	maximum 220 C	265.31	58.4	4.54	maximum 220 C	irradiated
6	0.45	1.13	0.398	maximum 450 C	0.52	2.36	0.22	maximum 450 C	no evaluation possible
7	0.43	4.17	0.103	maximum 450 C	0.45	20.94	0.021	maximum 450 C	untreated
8	120.17	14.94	8.04	maximum 245 C	109.28	49.11	2.42	maximum 245 C	irradiated
9	18.82	523.24	0.036	maximum 245 C	0.88	120.03	0.007	maximum 320 C	irradiated
10	0.98	52.73	0.019	maximum 310 C	0.48	150.25	0.003	maximum 450 C	no evaluation possible
11	629.05	300.62	2.09	maximum 245 C	211.19	73.41	2.87	maximum 245 C	irradiated
12	59.39	31.07	1.91	maximum 245 C	47.75	26.36	1.81	maximum 245 C	irradiated
13	0.4	0.76	0.526	maximum 450 C	0.39	6.66	0.059	maximum 450 C	untreated
14	0.54	58.19	0.009	maximum 420 C	0.58	78.83	0.007	maximum 420 C	untreated
15	695.29	77.25	9.00	maximum 245 C	1657.65	502.78	3.3	maximum 245 C	irradiated
16	0.42	81.86	0.005	maximum 450 C	0.39	29.18	0.013	maximum 450 C	untreated
17	85.9	4901.15	0.018	maximum 295 C	42.78	2145.06	0.02	maximum 295 C	irradiated
18	0.39	11.41	0.034	maximum 450 C	0.41	33.81	0.012	maximum 320 C	untreated

Table D.16 TL data for TL Laboratory 19

APPENDIX E

MIXING THE BLENDS

FSA PT SCHEME – MIXING OF BLENDS FOR ROUND 3 PSL AND TL

Products were irradiated at Isotron on 29/03/07 (10 herbs and spices and 5 bulk dietary supplements) and 6 chosen as the raw material for the blends for round 3. Products chosen for mixing were SP10895 (ground cinnamon), SP10897 (ground cumin), SP10902 (thyme), SP10950 (Siberian ginseng), SP10951 (alfalfa) and SP10954 (guarana). The material for these latter 3 was purchased in bulk, i.e.not ready potted. The other 3 products were purchased in cardboard containers of 2 cellophane sachets each, of known weight.

After the experience of round 2, it was decided not to conduct as many PSL measurements during the mixing process. Also on the basis of round 2 experience, mixing of the spike with the unirradiated matrix was performed by sprinkling followed by folding in with a spoon.

Since all these products were used in the previous 2 rounds, albeit from different batches and different irradiations, sensitivity estimates from earlier measurements were initially used to decide on the concentrations of spike. The 3 dietary supplements chosen differed in sensitivity by approximately an order of magnitude, and the same is true of the herb and spices, providing examples of high, medium and low sensitivity for each sub-set. Accordingly, the highest sensitivity products (thyme and ginseng) were chosen for the lowest (0.1%) concentration, the lowest sensitivity (cinnamon and guarana) for the highest concentration (10%) and the other two products for the intermediate concentration (1%).

6 aliquots from each of the end members, plus 6 from the blend, were measured over 60s for each product in turn as it was mixed. This took place for the dietary supplements on 03.04.07.

Starting this process with the ginseng, it was discovered that this product, which had arrived mis-labelled, had a totally different, extremely low, sensitivity from the very high sensitivity batch used in rounds 1 and 2. It was therefore decided to substitute SP10952 (green tea) which had a medium sensitivity similar to the guarana. The new alfalfa had the highest sensitivity of the 3, so was used for the 0.1% mix. Blending was performed using the bulk material as purchased for the matrix, and an irradiated sub-sample of the bulk for the spike.

At this point is was discovered that the “unirradiated” alfalfa had an intermediate signal, as did the guarana and saw palmetto (not used in Round 3). Unirradiated green tea was negative.

The following self-blends were then decided upon : 0.1% alfalfa, 1% green tea and 10% guarana. These were made up from the bulk materials and decanted into 50 pots, lids put on and labels applied.

PSL data are as follows:

	Unirradiated	Spike	Blend
Aliquot			
1	3027	2153042	8815
2	3357	1967392	3870
3	3642	1764246	3471
4	1937	1506443	14635
5	1828	1346770	3408
6	2105	1566491	8718
Mean	2649	1717397	7153
SD	788	303024	4462
CV(%)	30	18	62

Alfalfa 0.1% (1g in 1000g)

	Unirradiated	Spike	Blend
Aliquot			
1	579	37183	905
2	539	29600	1626
3	559	23394	801
4	523	46657	2998
5	501	25308	995
6	558	43007	783
Mean	543	34192	1351
SD	28	9577	865
CV(%)	5	28	64

Green tea 1% (10g in 1000g)

	Unirradiated	Spike	Blend
Aliquot			
1	480	20899	1933
2	391	12307	1762
3	438	8020	1657
4	464	11158	959
5	457	28642	1575
6	495	41708	2702
Mean	454	20456	1765
SD	37	12856	566
CV(%)	8	63	32

Guarana 10% (100g in 1000g)

Table E.1 PSL screening data for mixing of alfalfa, green tea and guarana

On 04.04.07 the second batch of irradiated materials arrived back from Isotron. The ginseng in that batch (already potted by CCL) contained a substantial PSL signal and is therefore a) not the same material as the bulk and b) suitable for the positives in the trial. This removed the need to pot the bulk irradiated green tea, of which there was not really enough to provide positive samples for all laboratories plus reference analysis.

A single aliquot of each of the chosen herb and spices was checked, confirming the relative sensitivities. The concentrations were therefore as previously allocated.

For the thyme, the high volume to weight ratio lead to a decision to make up a total of 700g (0.7g spike). Pots 71-128 were used for the unirradiated portion, and pot 2 for the spike.

	Unirradiated	Spike	Blend
Aliquot			
1	515	2410216	10131
2	500	2205707	1496
3	452	2158395	2675
4	479	1960452	4765
5	484	2236939	11460
6	430	2384484	3389
Mean	477	2226032	5653
SD	31	164116	4143
CV(%)	7	7	73

Thyme 0.1% (0.7g in 700g)

For the cumin, 1kg was prepared (10g from pot 64 and 990g from pots 73-109).

	Unirradiated	Spike	Blend
Aliquot			
1	337	47930	1194
2	407	43333	5110
3	393	43117	2449
4	441	70532	1176
5	337	46936	1393
6	373	47813	780
Mean	381	49944	2017
SD	41	10313	1616
CV(%)	11	21	80

Cumin 1% (10g in 1000g)

For the cinnamon, 1kg was prepared (100g from pots 13,17,18 and 40 and 900g from pots 72-98)

	Unirradiated	Spike	Blend
Aliquot			
1	320	1680	379
2	243	8936	472
3	376	1681	455
4	287	1742	454
5	325	1518	457
6	267	1105	648
Mean	303	2777	478
SD	47	3026	90
CV(%)	16	109	19

Cinnamon 10% (100g in1000g)

Table E.2 PSL screening data for mixing of thyme, cumin and cinnamon

33 pots of paprika standard were then dispensed and labelled for the labs doing PSL.

Boxes then made up ready for packing, after allocation of lab numbers based on order of reply. PSL labs were given sequence 1-33, TL labs 1-17; some labs may have 2 numbers if they are doing both techniques. Later 4 further PSL sets and 2 further TL sets were dispatched.

Sample number allocations were as follows:

Sample	Description	Status	Number	Sample no.
SP10951	Alfalfa	U	1	10
SP10951	Alfalfa	I	2	11
SP10951	Alfalfa	B	3	4
SP10950	Ginseng	U	4	17
SP10950	Ginseng	I	5	1
SP10952	Green tea	B	6	9
SP10954	Guarana	U	7	14
SP10954	Guarana	I	8	15
SP10954	Guarana	B	9	12
SP10902	Thyme	U	10	16
SP10902	Thyme	I	11	2
SP10902	Thyme	B	12	18
SP10897	Cumin	U	13	3
SP10897	Cumin	I	14	8
SP10897	Cumin	B	15	6
SP10895	Cinnamon	U	16	13
SP10895	Cinnamon	I	17	5
SP10895	Cinnamon	B	18	7

Table E.3 Sample allocations for Round 3 (PSL and TL)